Supporting Information

$FeCl_3$ Catalyzed Synthesis of Fluorene-C-9-linked Furan Hybrids from Biphenyl-linked Conjugated Ene-Yne-Ketones

Asadujjaman Nur, Abhishek Kar, Kush Mandal, Raj Kamal Sahoo, and Umasish Jana*.

Department of Chemistry, Jadavpur University, Kolkata 700 032 West Bengal, India

Corresponding Author Contact: E-mail: jumasish2004@yahoo.co.in

umasish@gmail.com

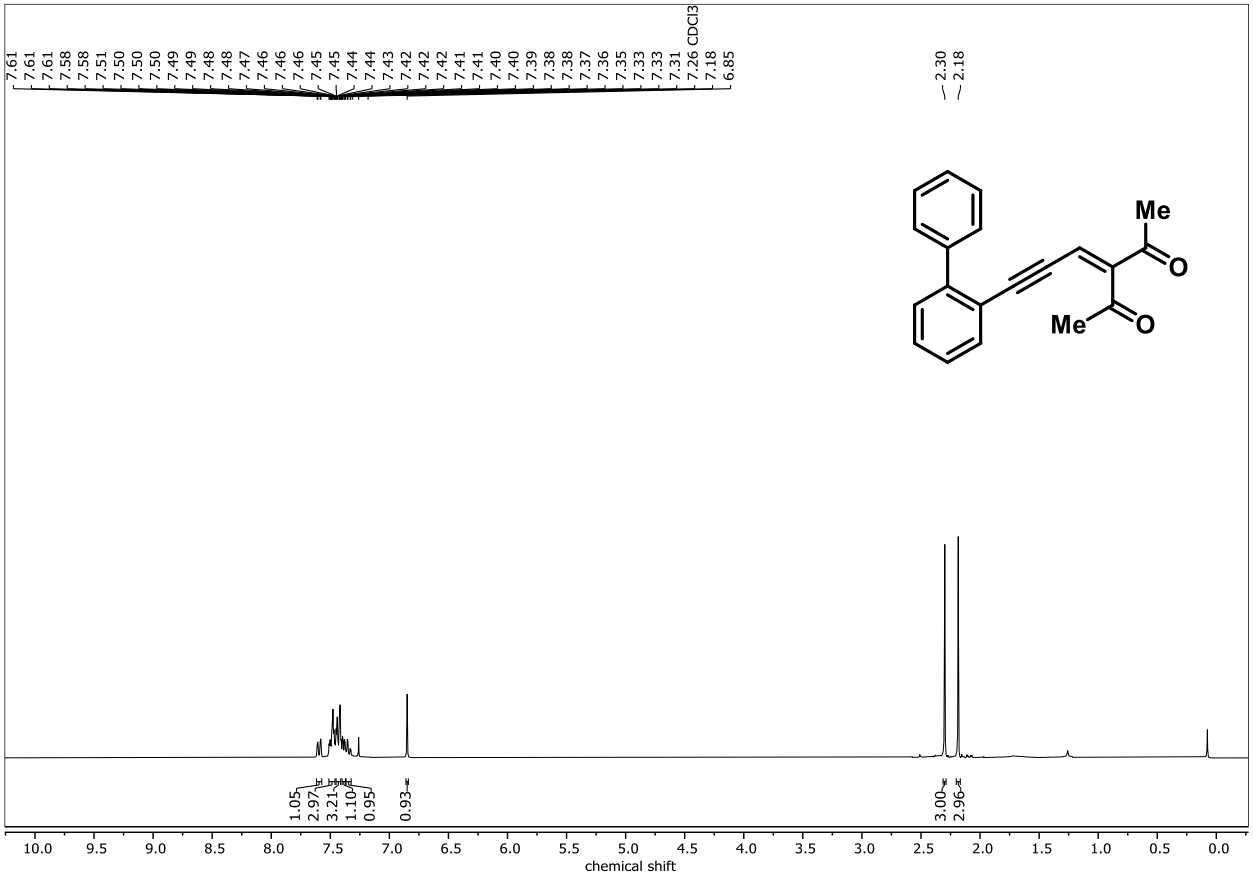
Table of Content

General Information. Unless otherwise mentioned, all reactions were carried out in oven-dried glass reaction vessels with magnetic stirring under argon atmosphere. Solvents, reagents, and chemicals purchased from Aldrich, Alfa Aesar, Merck, SRL, Spectrochem, and Process Chemicals were all used without further purification. The routine monitoring of the reactions was performed using analytical thin layer chromatography (TLC) employing Merk® silica gel 60 F254 plates and all chromatographic purifications were performed using Merk® silica gel (60–120 mesh).

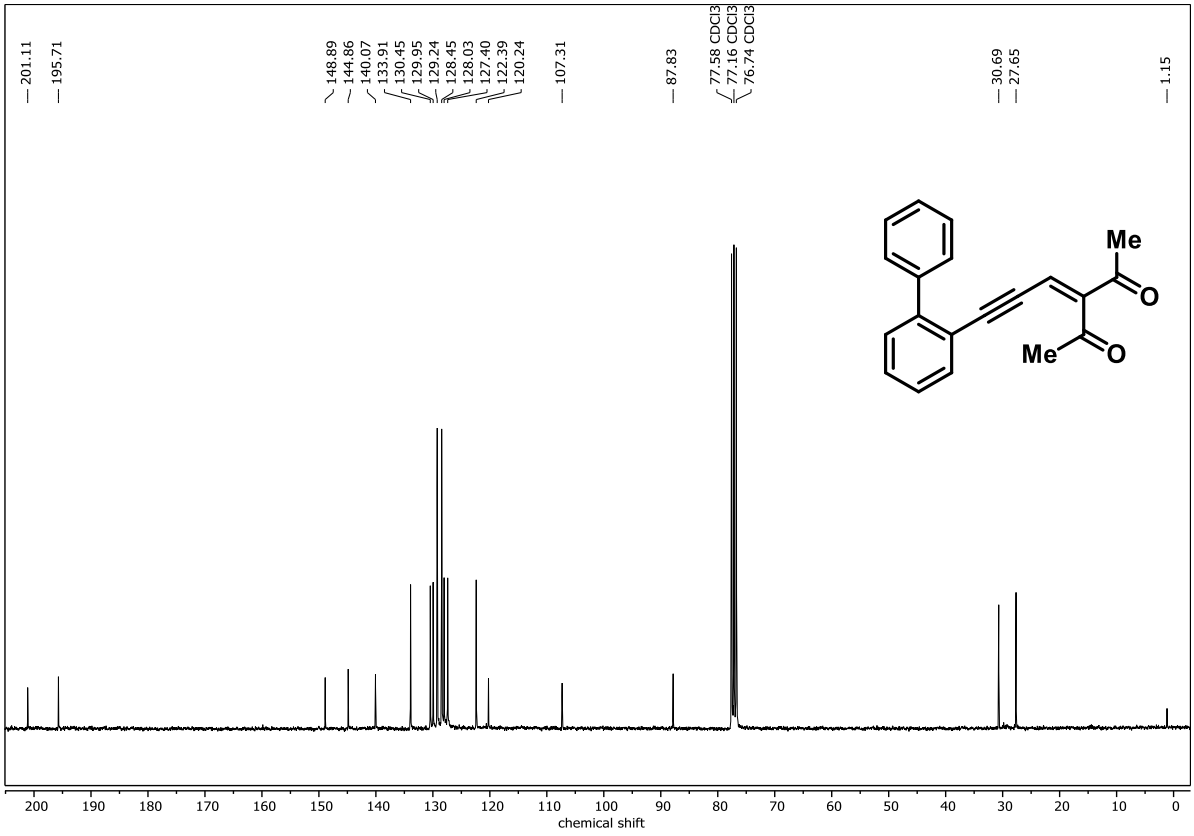
All ^{1}H NMR spectra were recorded with Bruker Avance III 300 (300MHz) and Bruker Avance III 400 (400MHz) spectrometers in deuterated solvent – $CDCl_3$. Chemical shifts are reported in parts per million (ppm, δ) relative to tetramethylsilane (TMS), and the solvent resonances were referenced to internal standard $CDCl_3$ (δ 7.26 ppm). The multiplicity descriptions of the signals are reported as follows: s = singlet, d = doublet, dd = double of doublet, t = triplet, m = multiplet, and dt = doublet of triplets. $^{13}C\{^{1}H\}$ NMR spectra were recorded with Bruker Avance III 300 (75 MHz) and 400 (101 MHz) spectrometers as solutions in $CDCl_3$ with complete proton decoupling. Chemical shifts are reported in parts per million (ppm, δ) and are referenced to internal standard $CDCl_3$ (δ 77.16 ppm). All coupling constants are absolute values and are expressed in Hz. High-resolution mass spectra were acquired using a Q-Tof Micro YA263 spectrometer in acetonitrile solvent and employing an electrospray ionization (ESI) technique. Crystallographic data were collected at room temperature on a Bruker APEX III D8 Quest smart diffractometer, equipped with a microfocus and a sealed tube X-ray source with graphite mono-chromated Mo-Kα radiation (λ = 0.71073 Å). All melting points were determined in a capillary melting point apparatus and are uncorrected. Infrared spectra of all the products (3a–3p) were recorded in the range 4000–400 cm^{-1} with a PerkinElmer FT-IR spectrometer (spectrum two).

^{1}H, $^{13}C\{1H\}$, and ^{19}F – NMR Spectra of Synthesized Products:

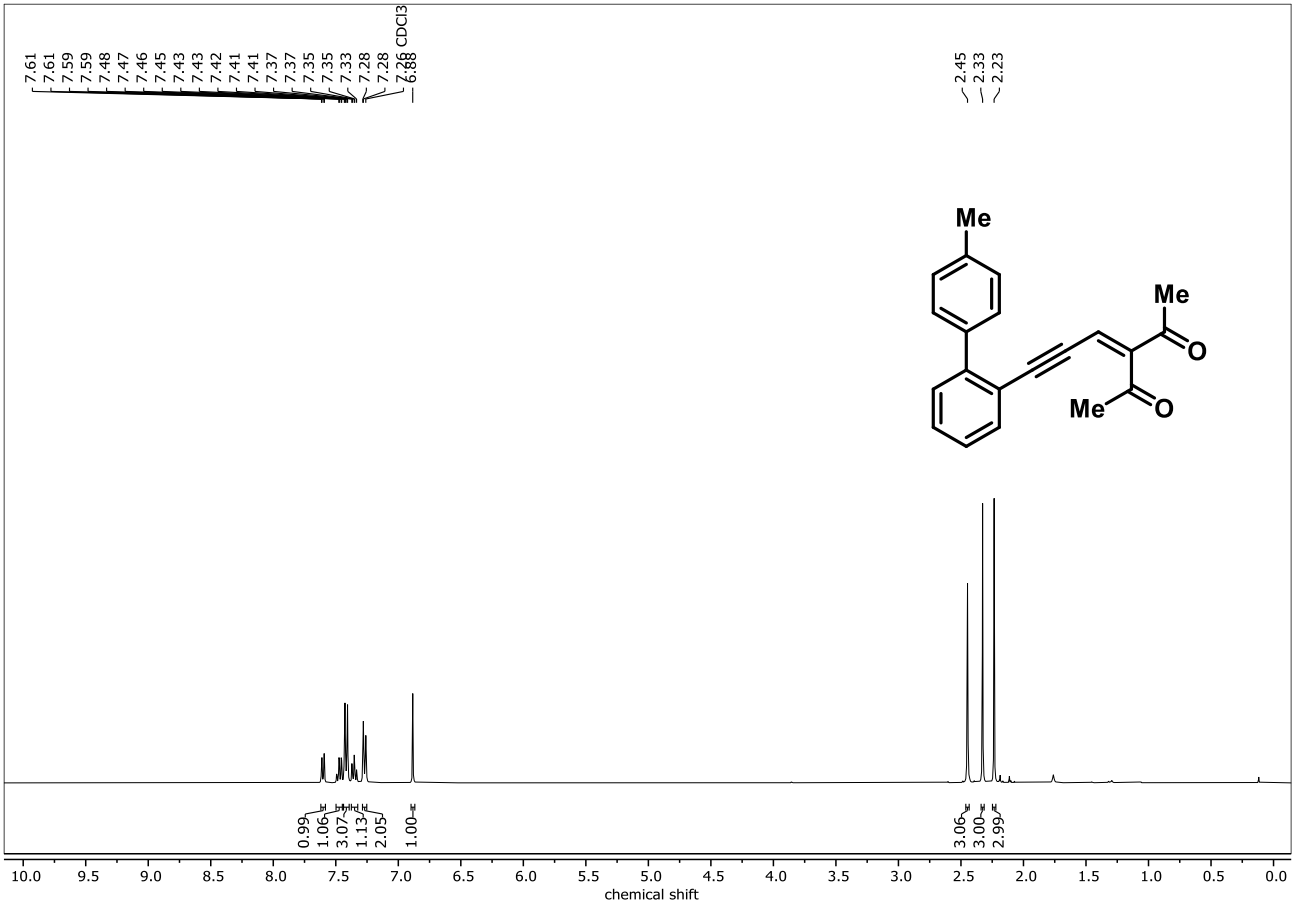
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2a



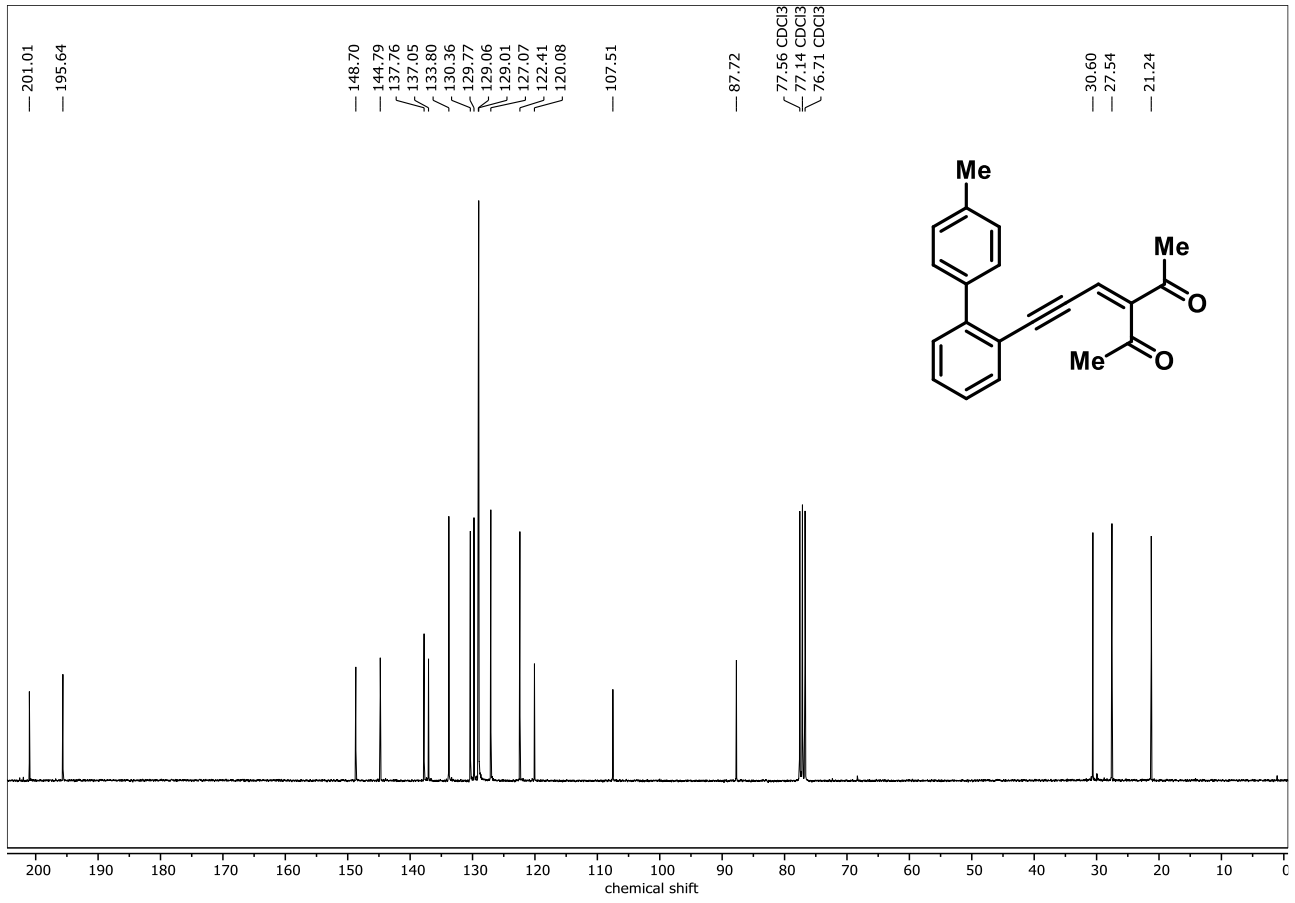
$^{13}C\{^{1}H\}$ (75 MHz, $CDCl_3$) NMR Spectrum of 2a



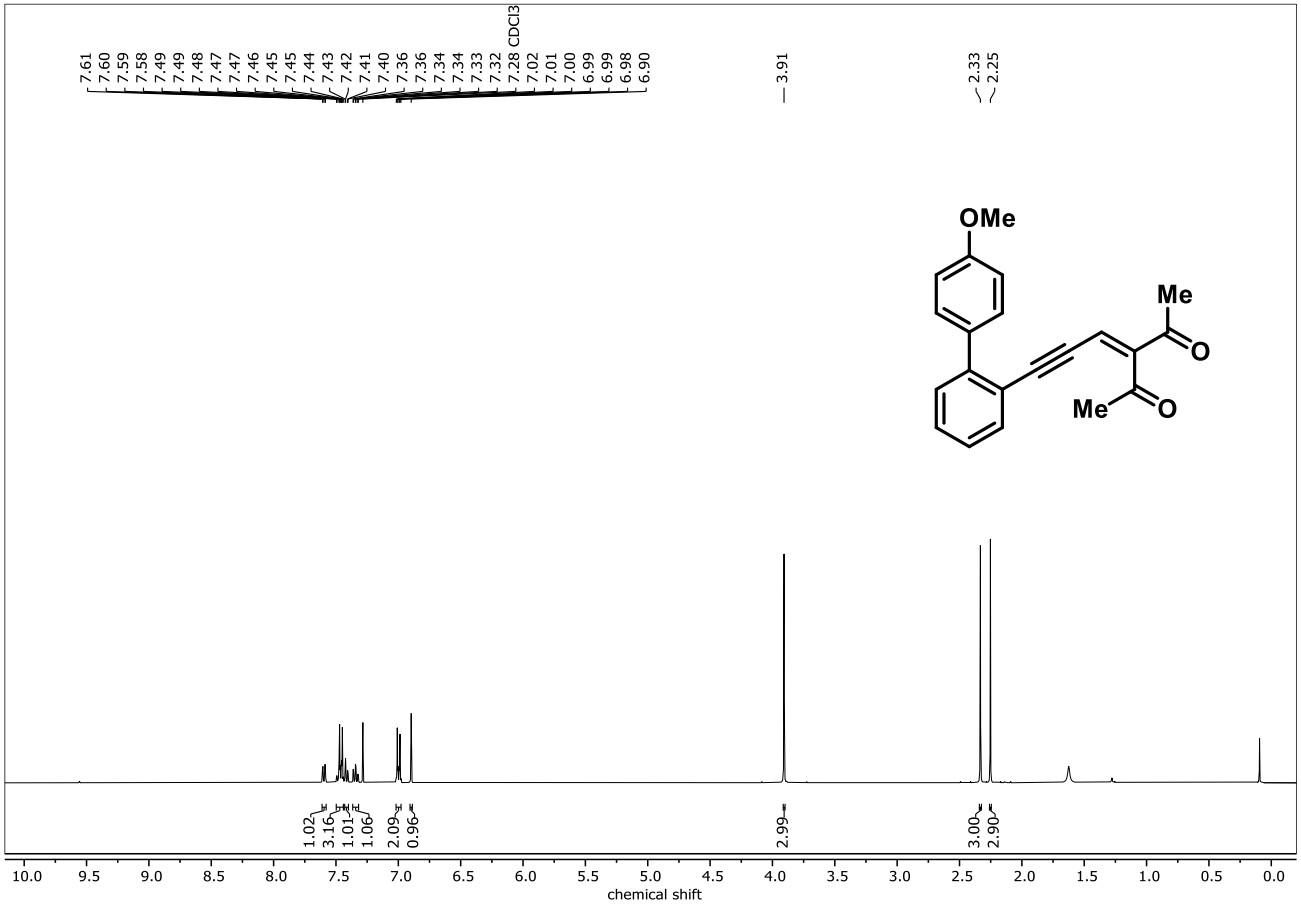
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2b



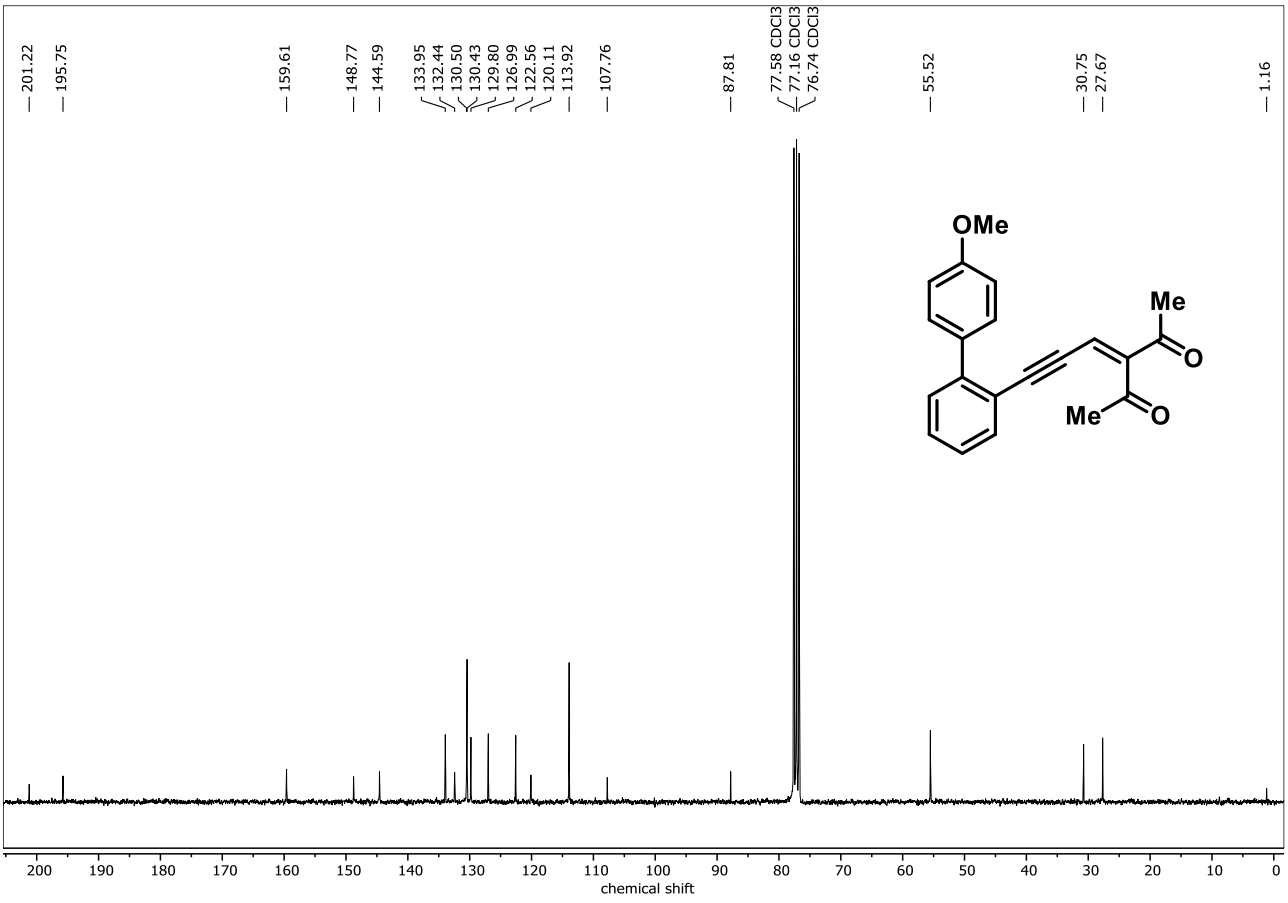
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2b



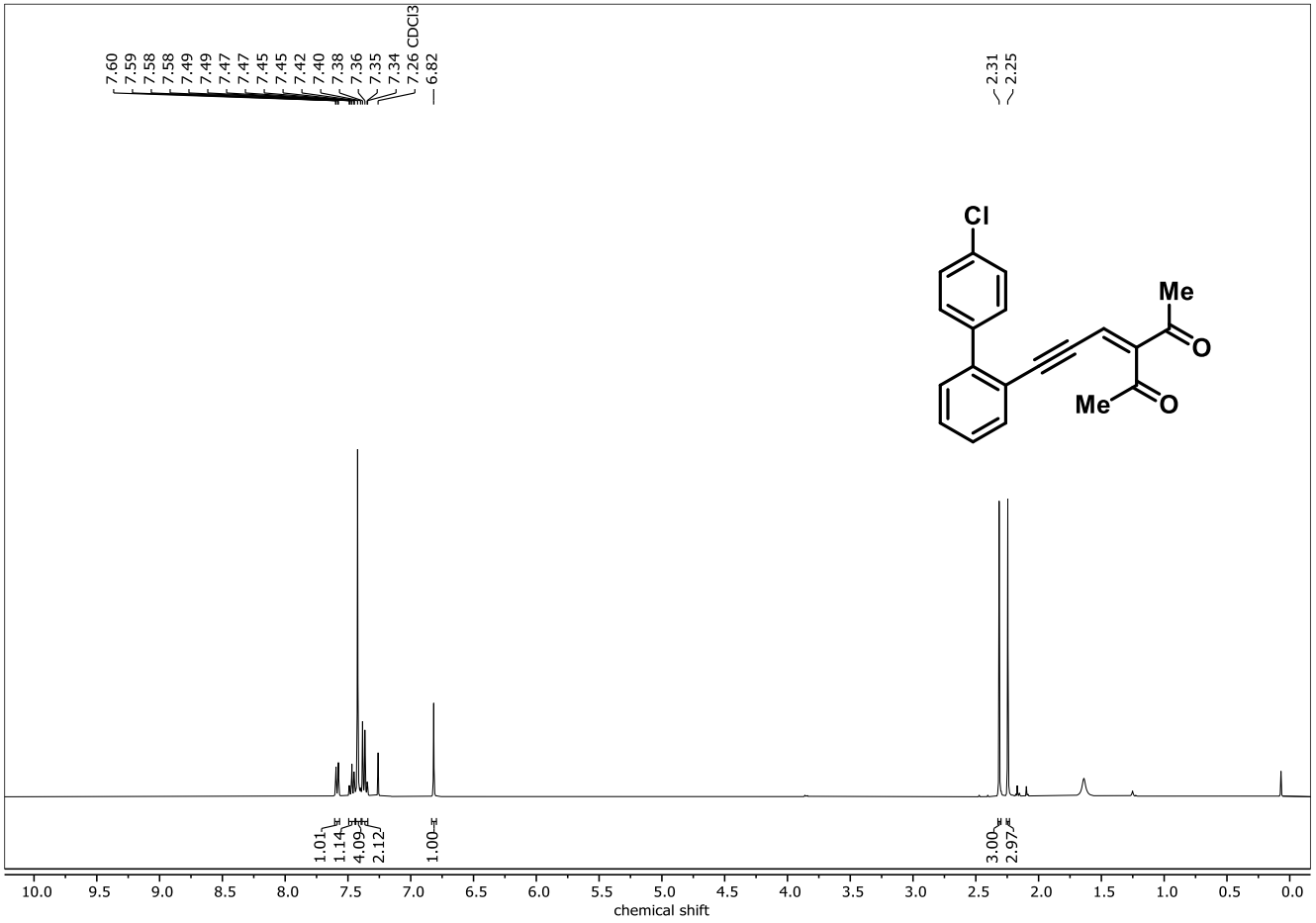
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2c



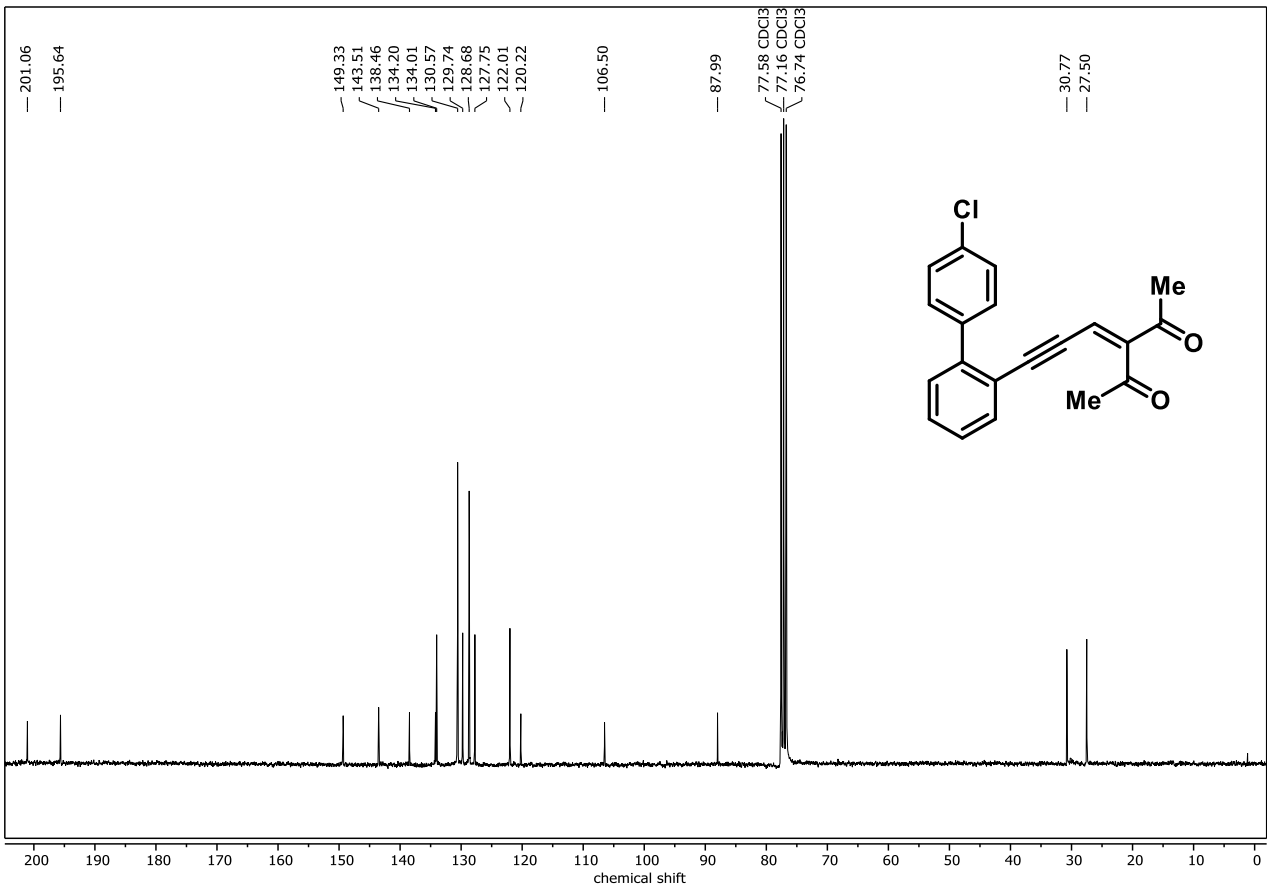
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2c



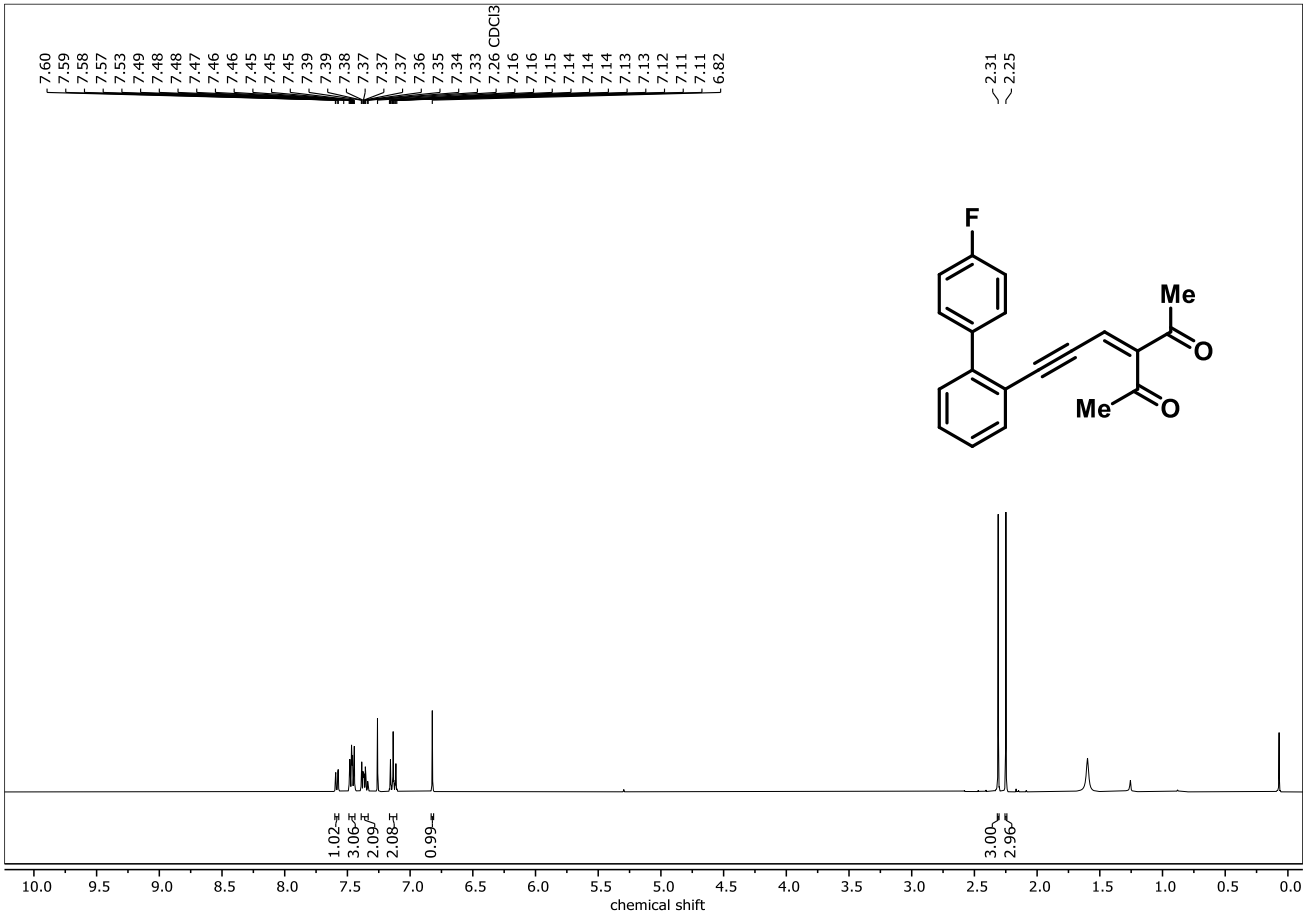
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2d



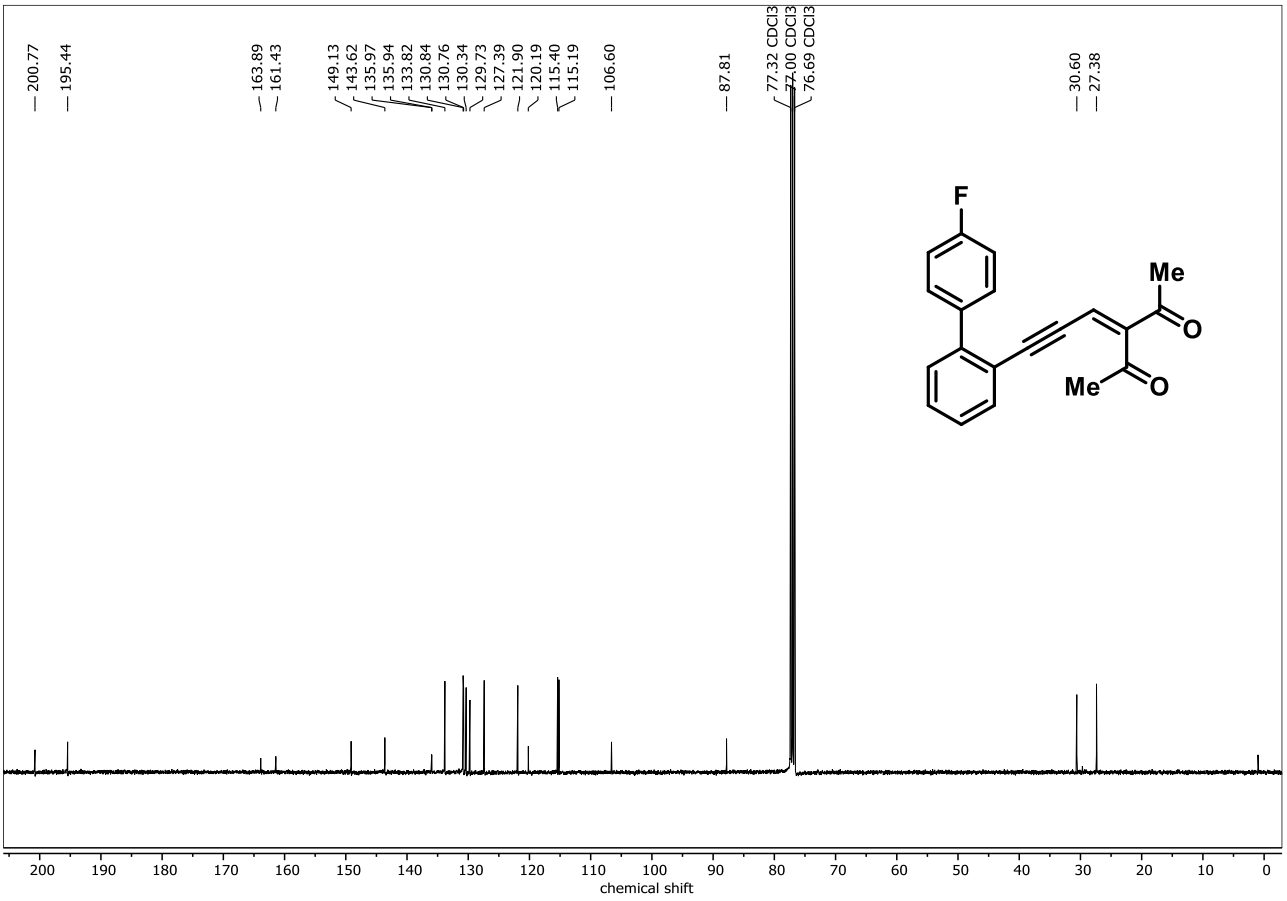
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2d



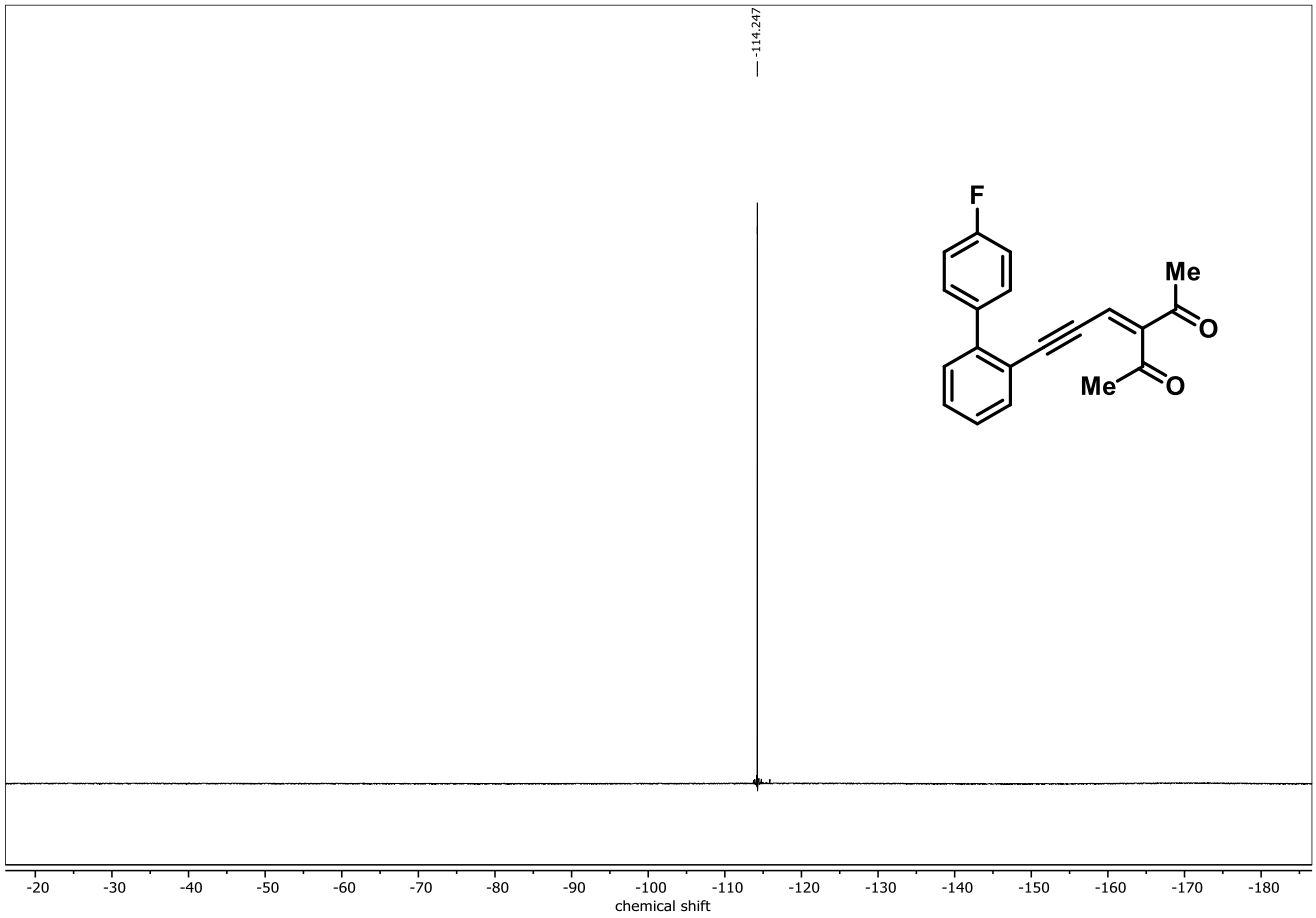
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2e



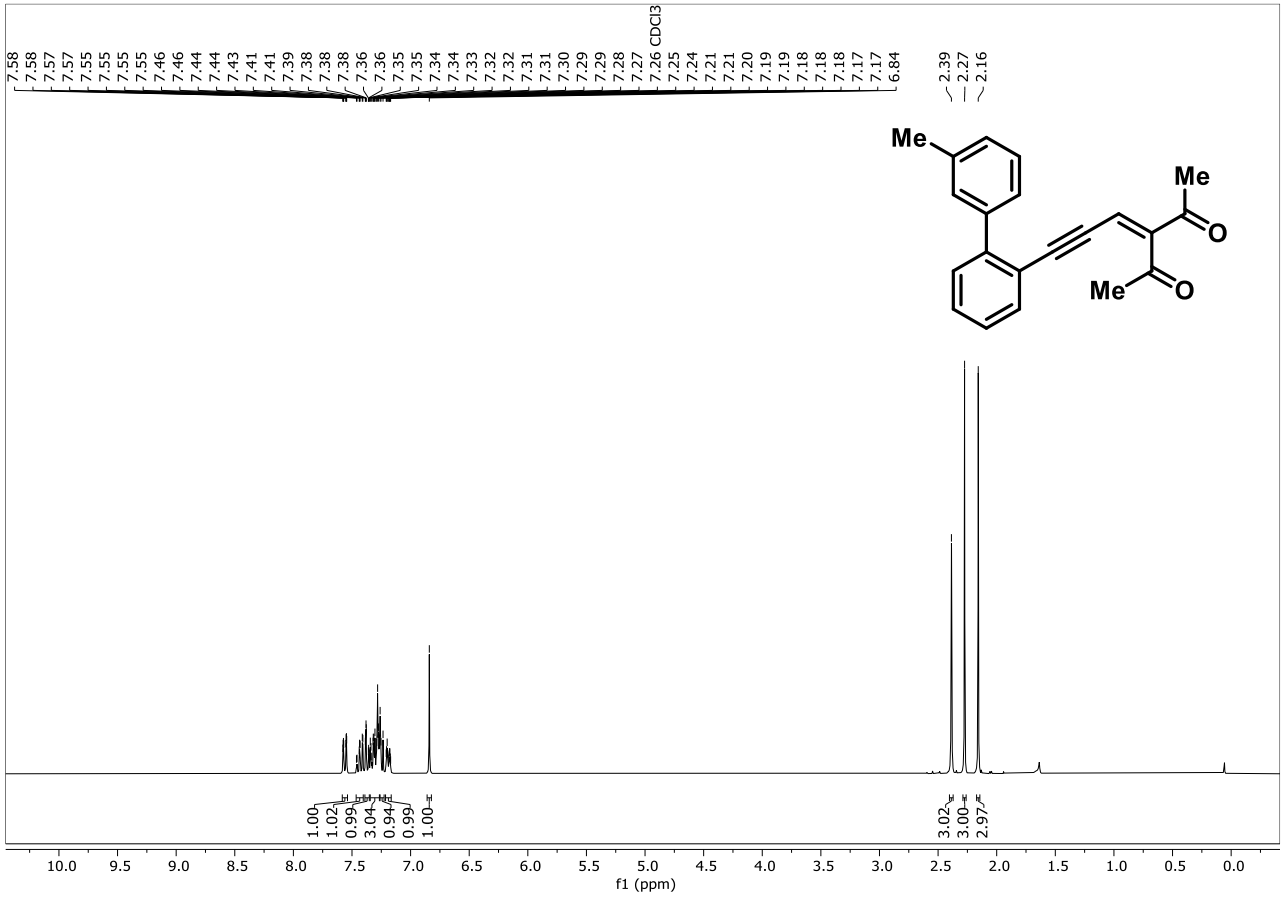
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 2e



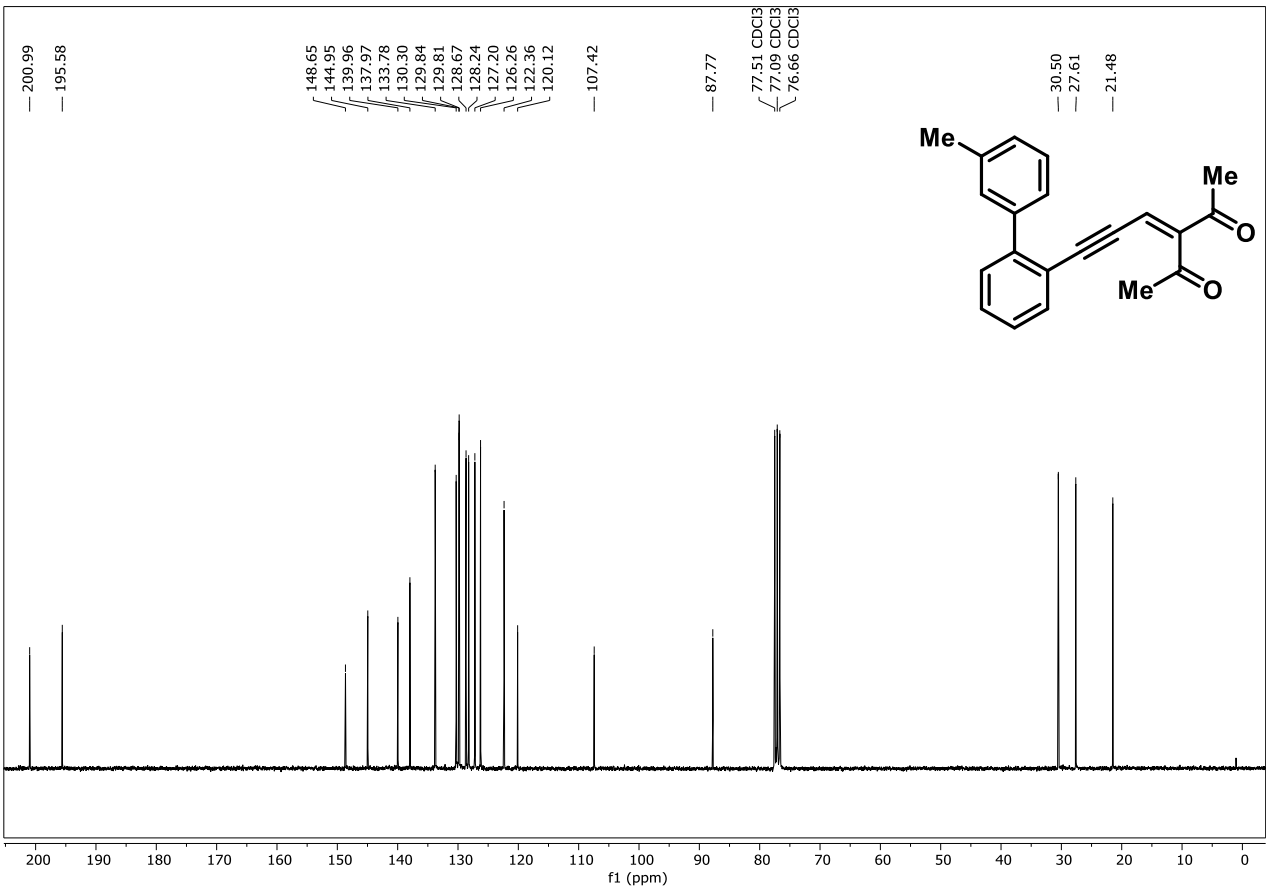
^{19}F (282 MHz, $CDCl_3$) NMR Spectrum of 2e



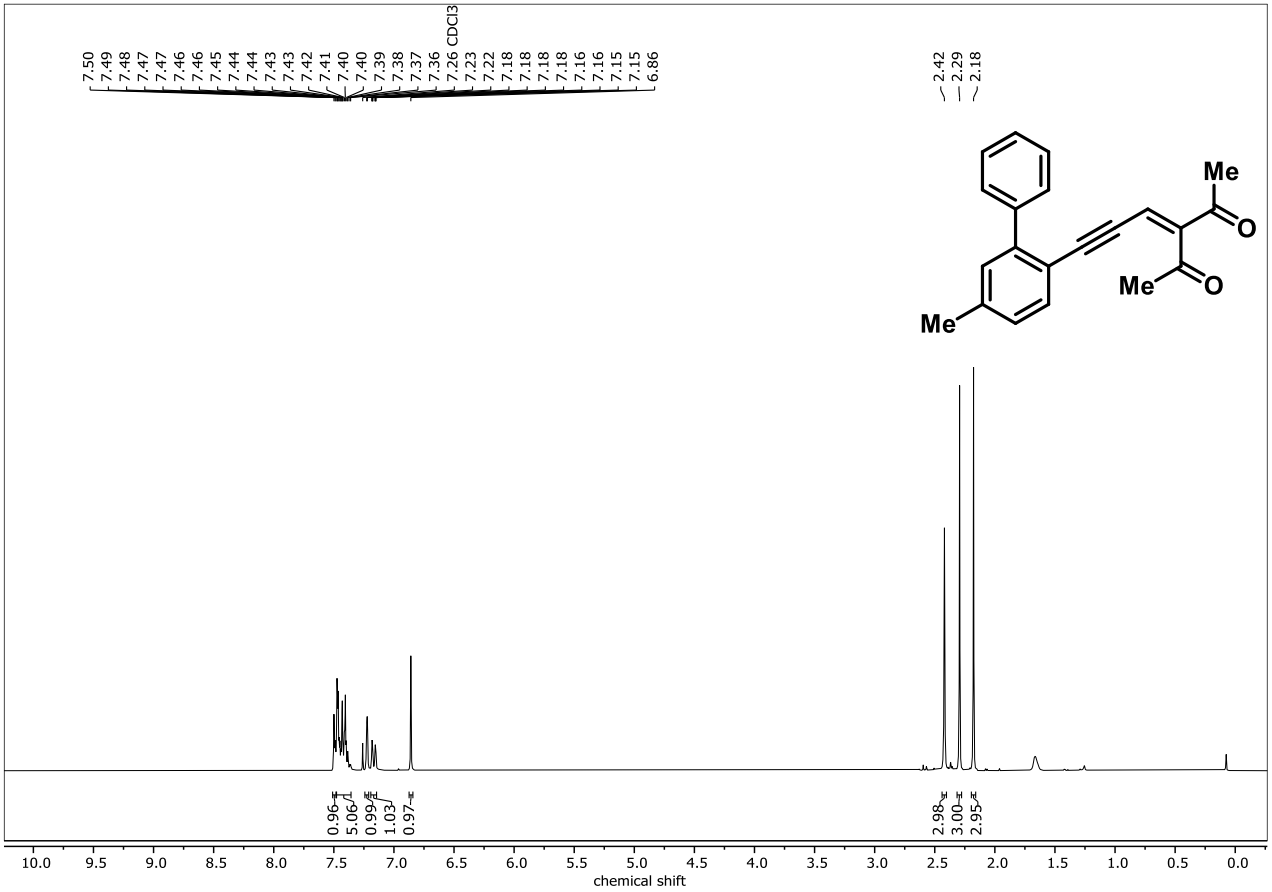
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2f



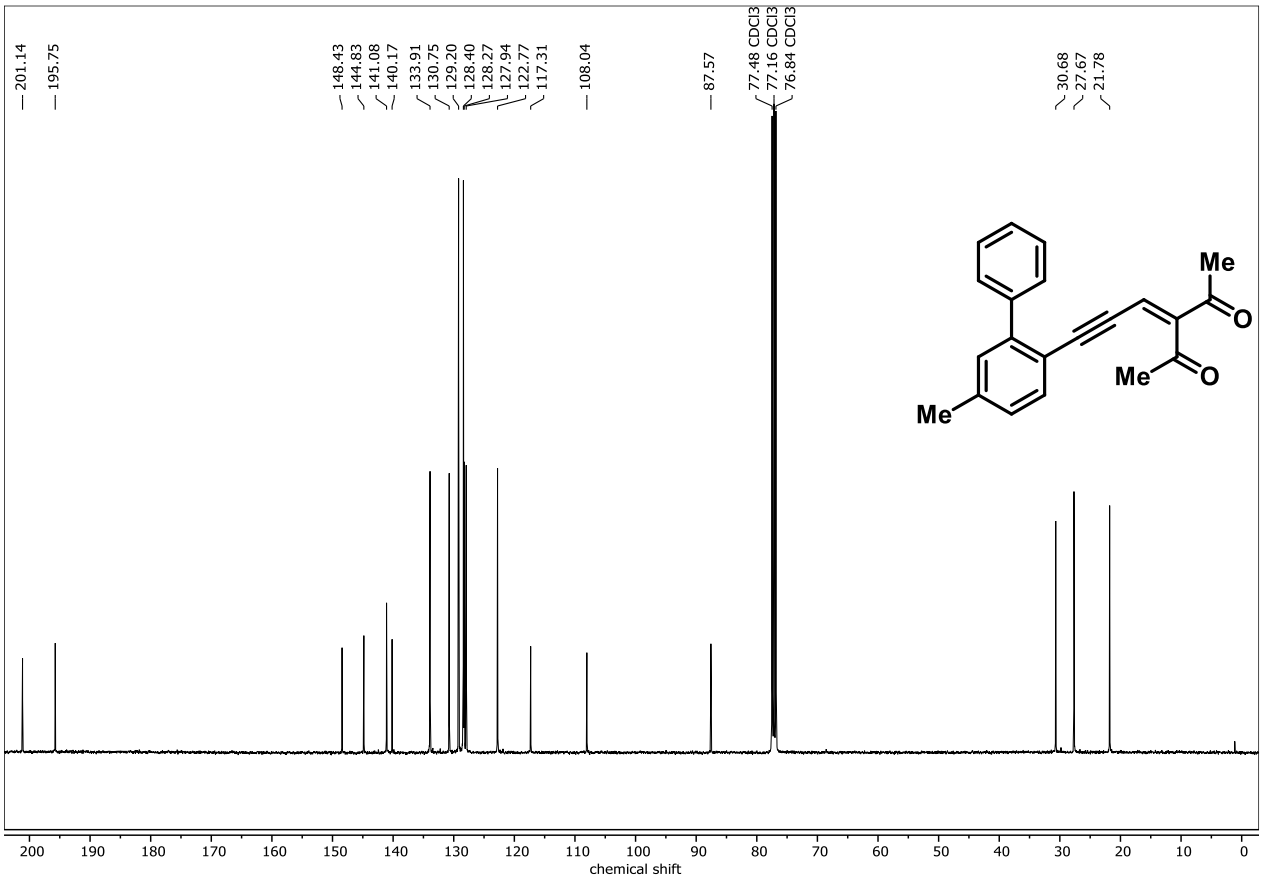
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2f



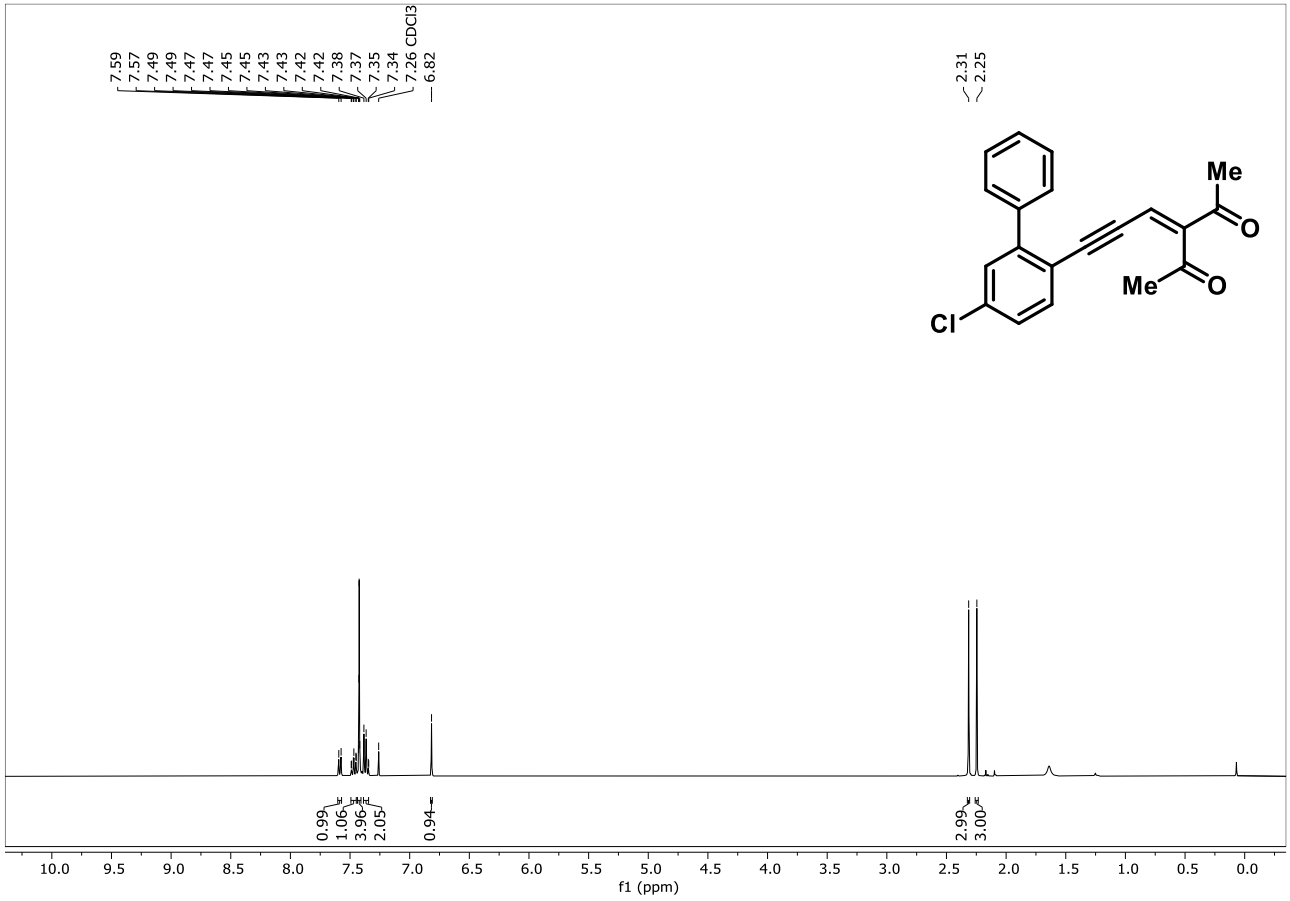
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2g



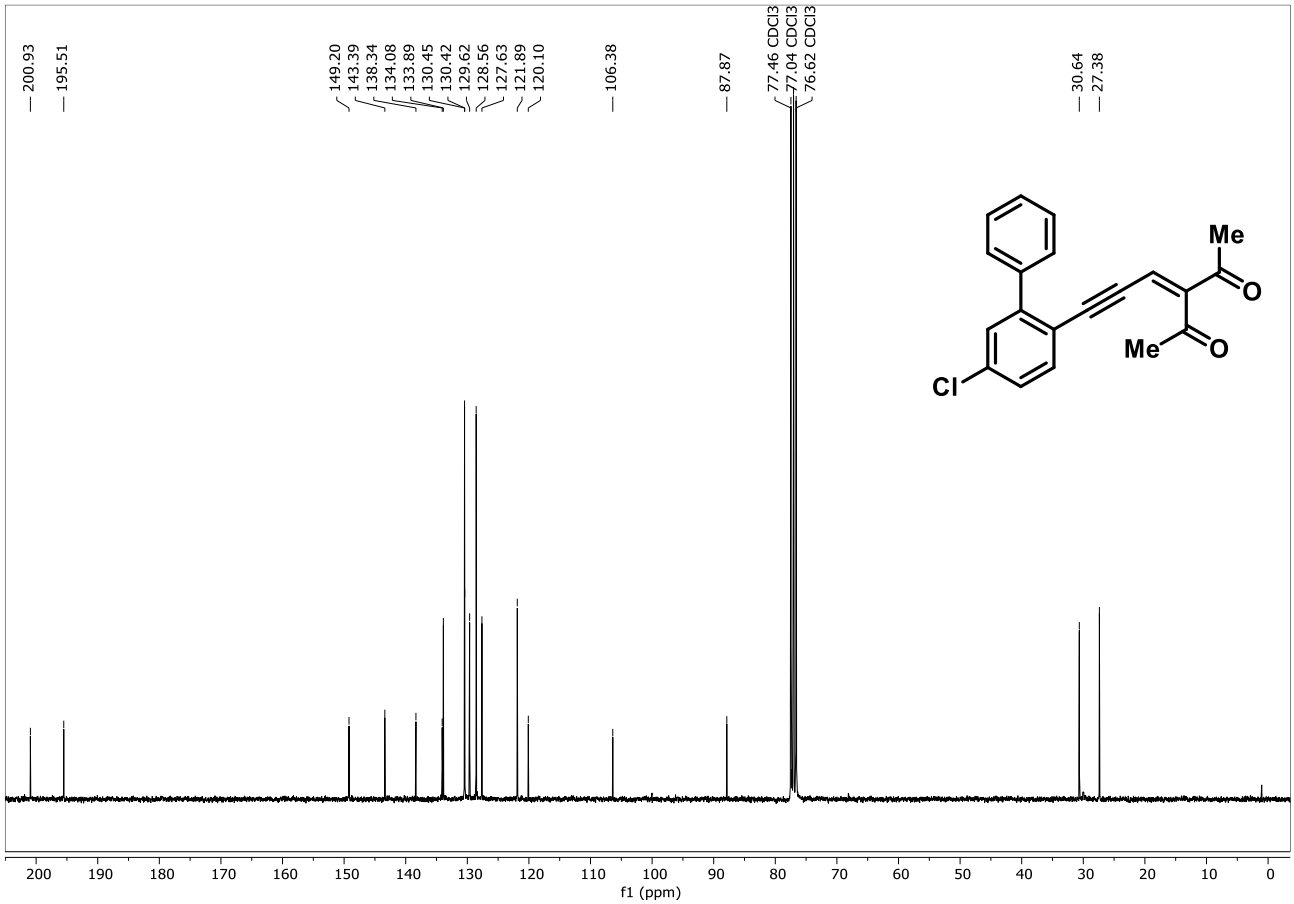
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 2g



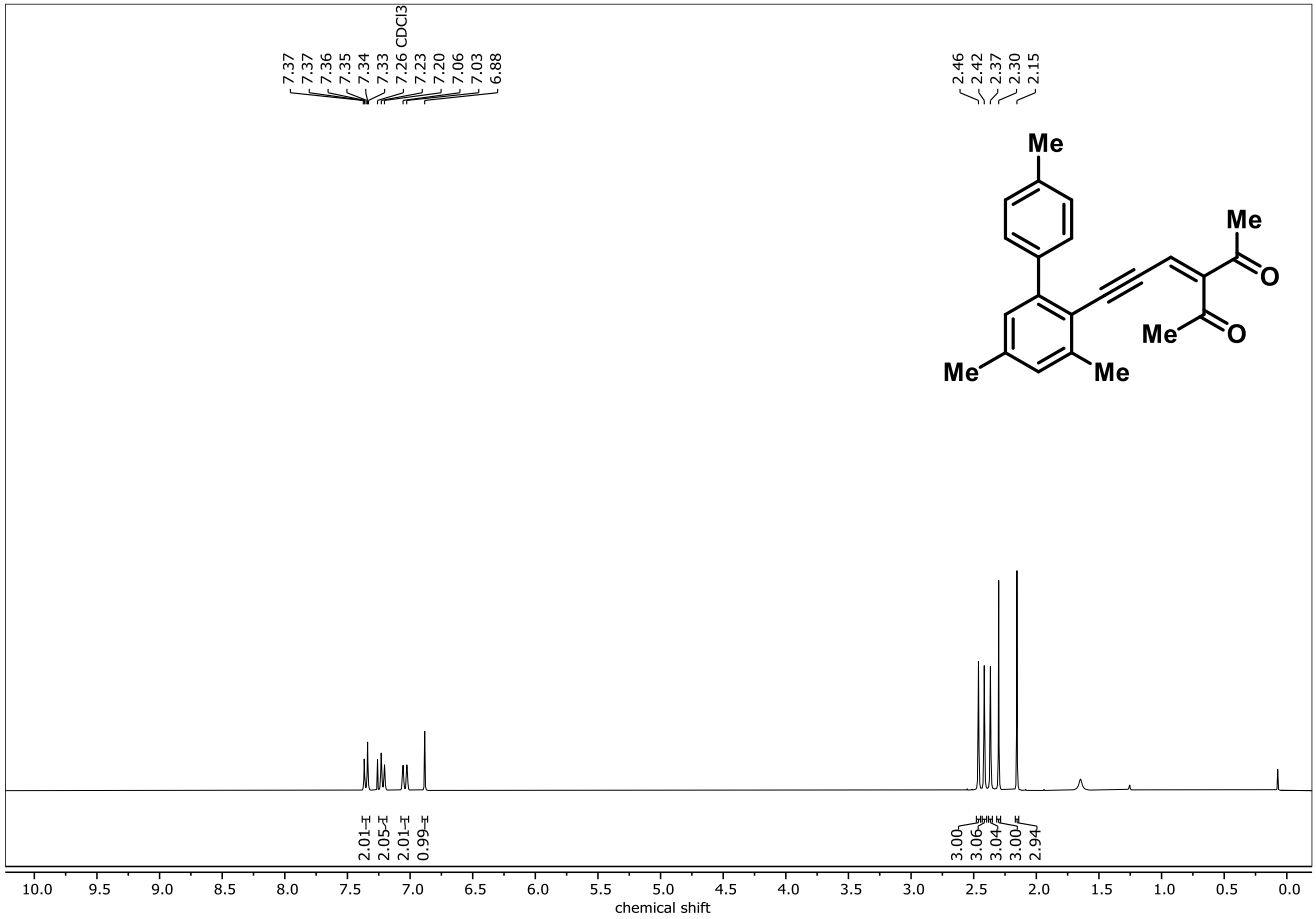
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2h



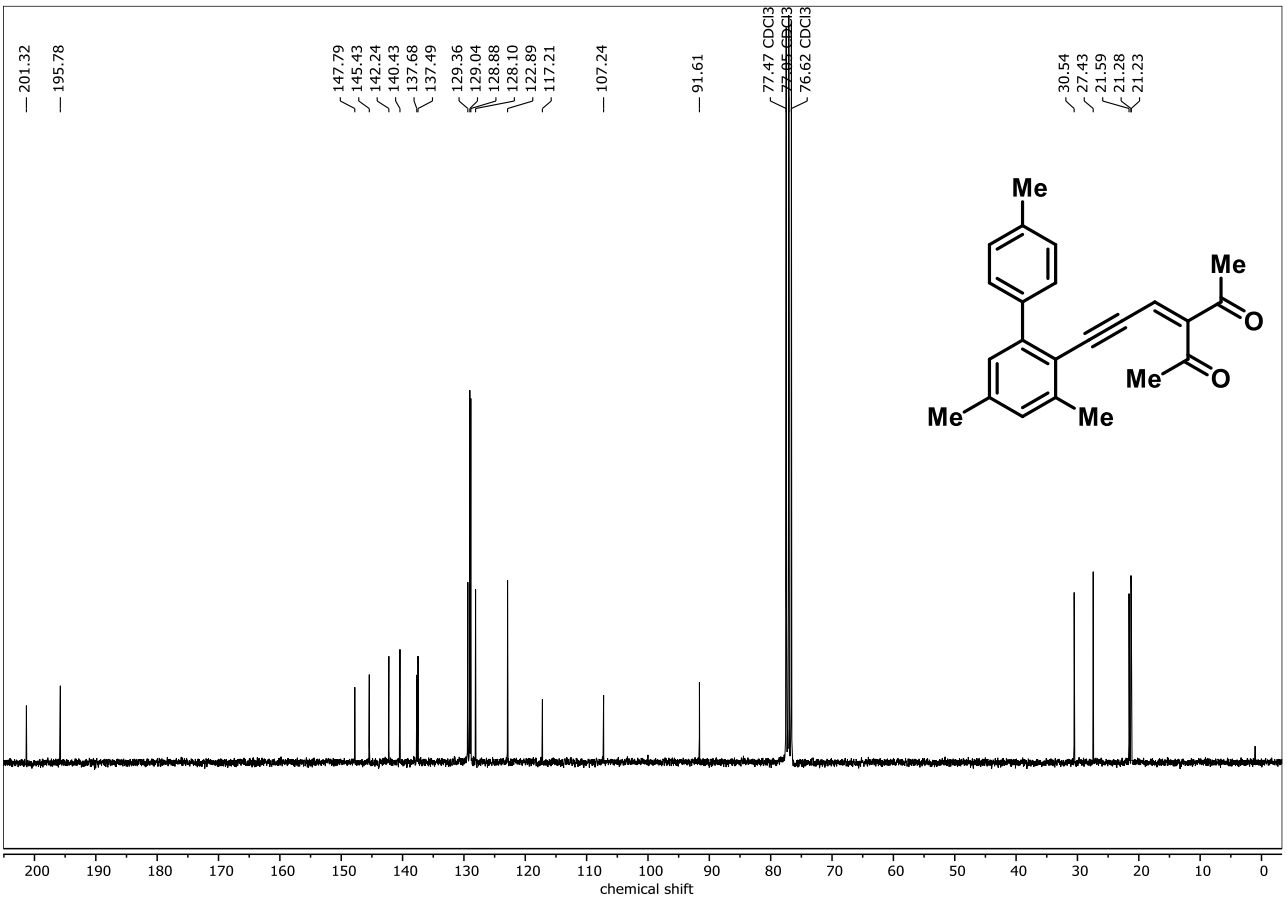
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2h



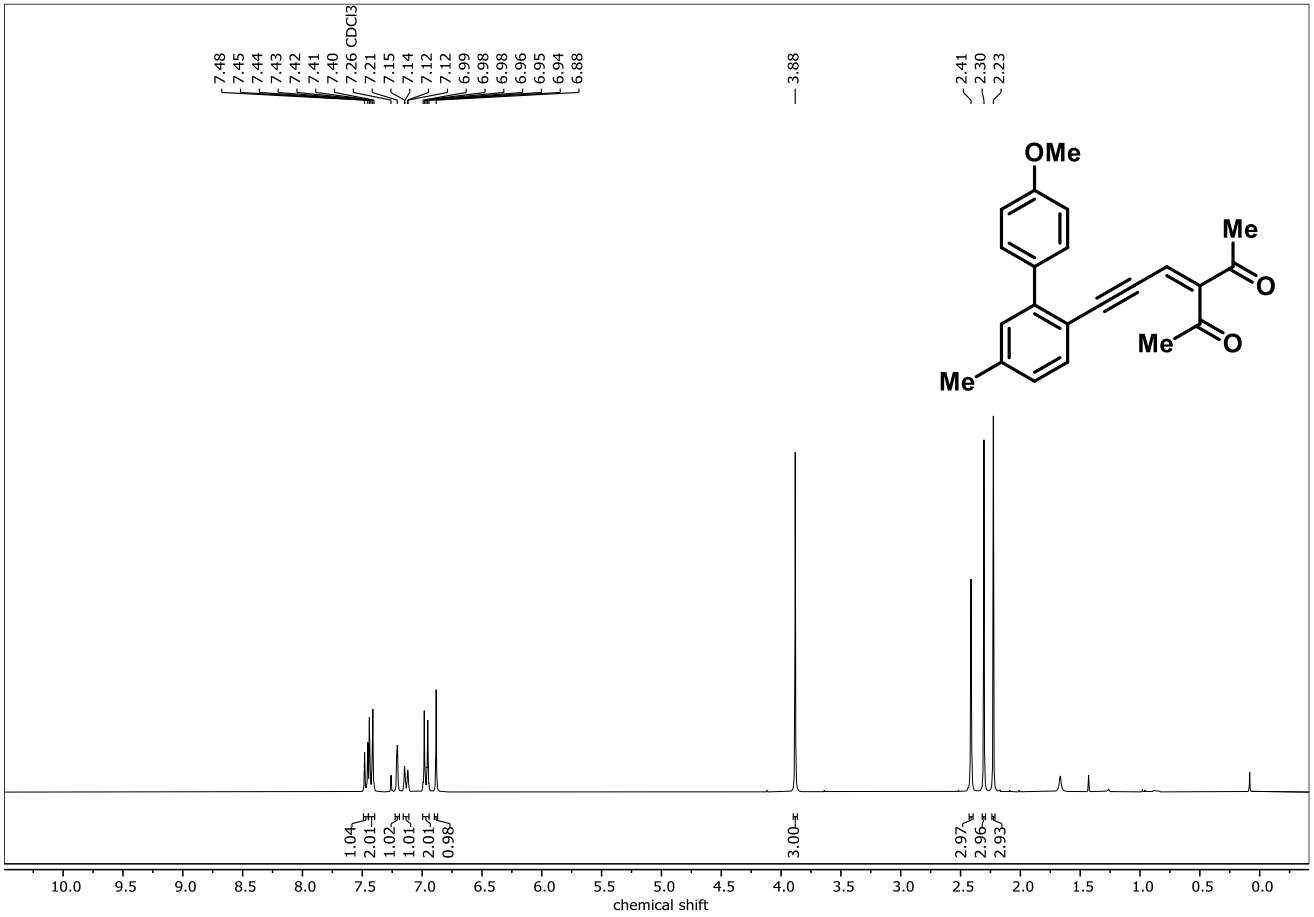
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2i



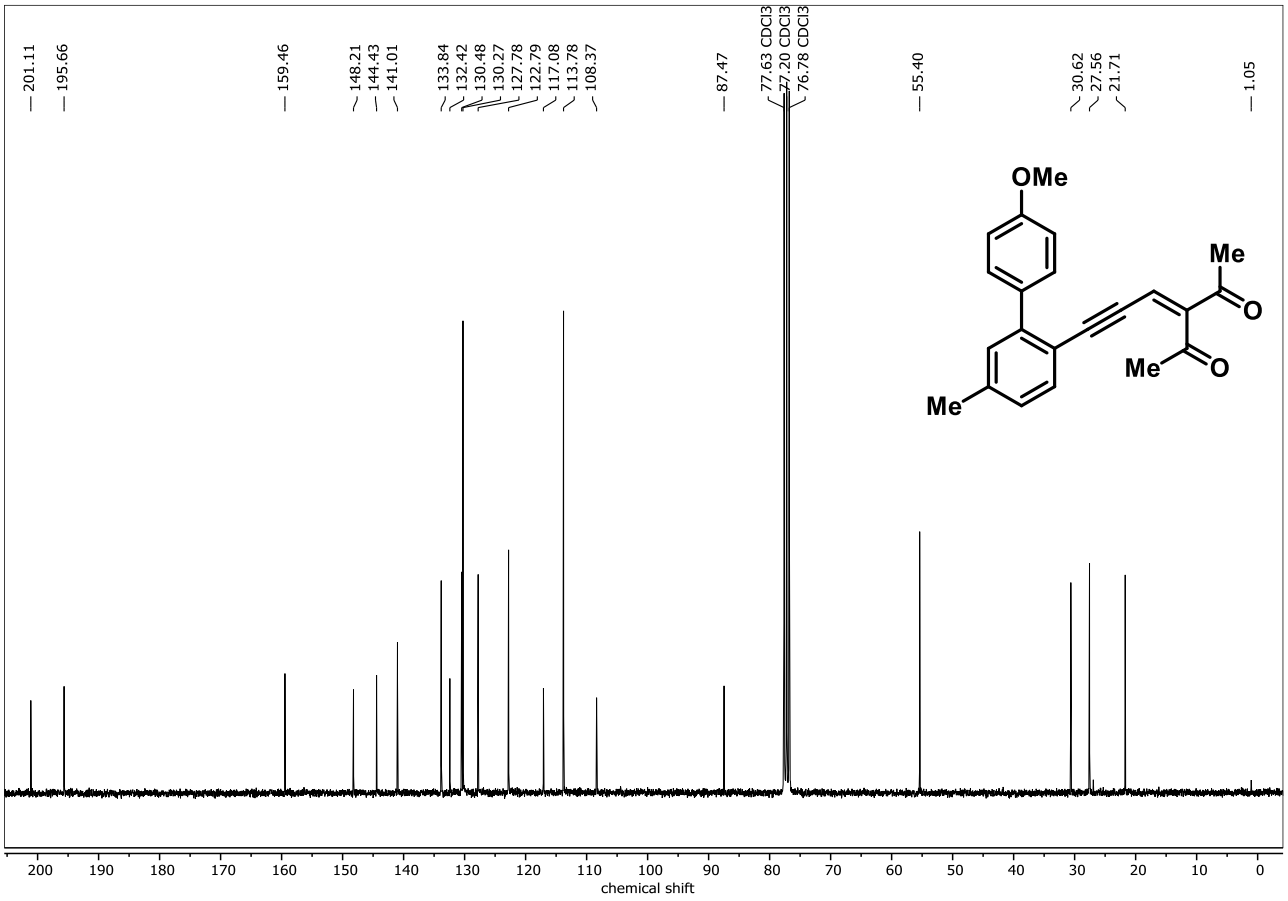
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2i



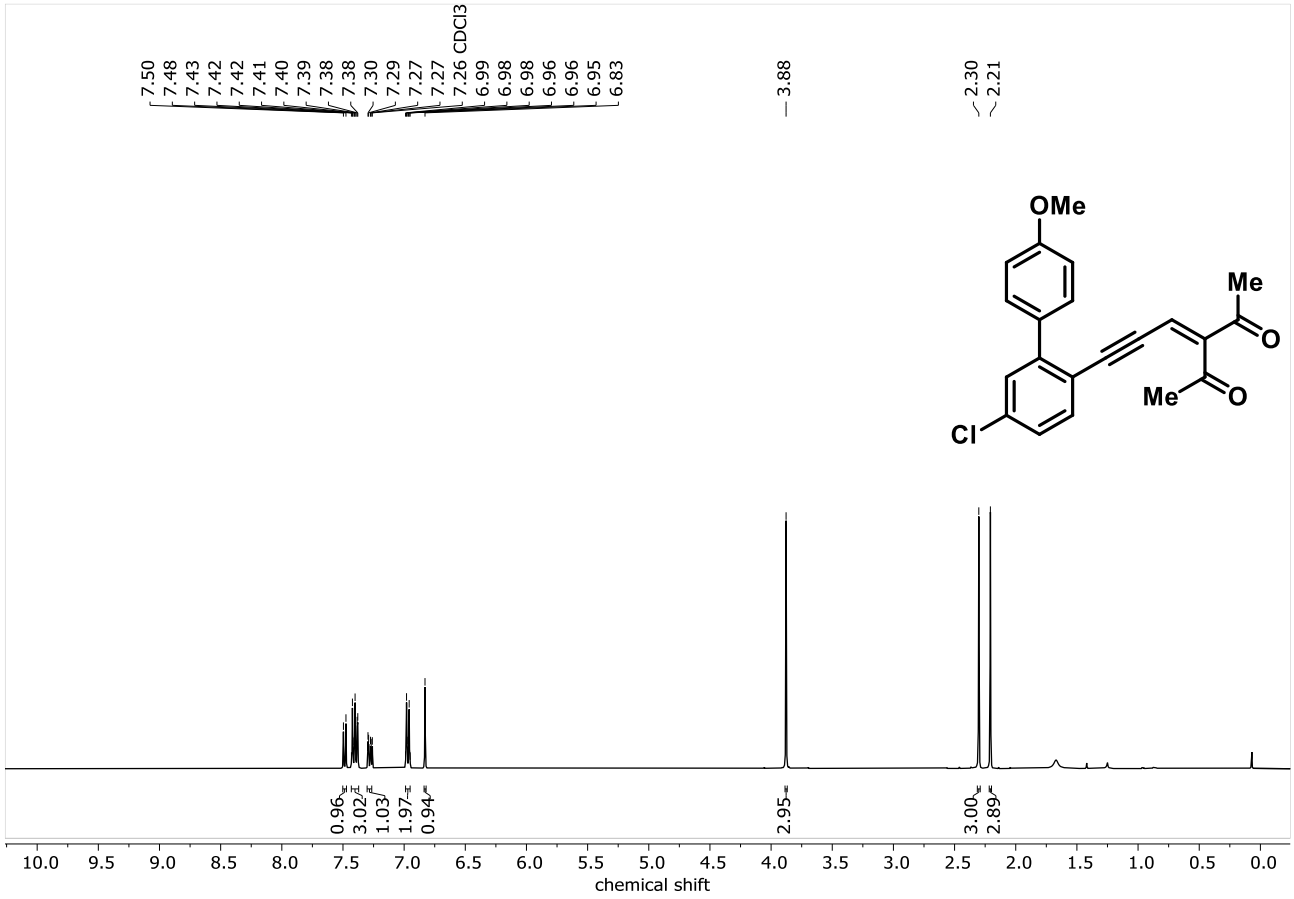
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2j



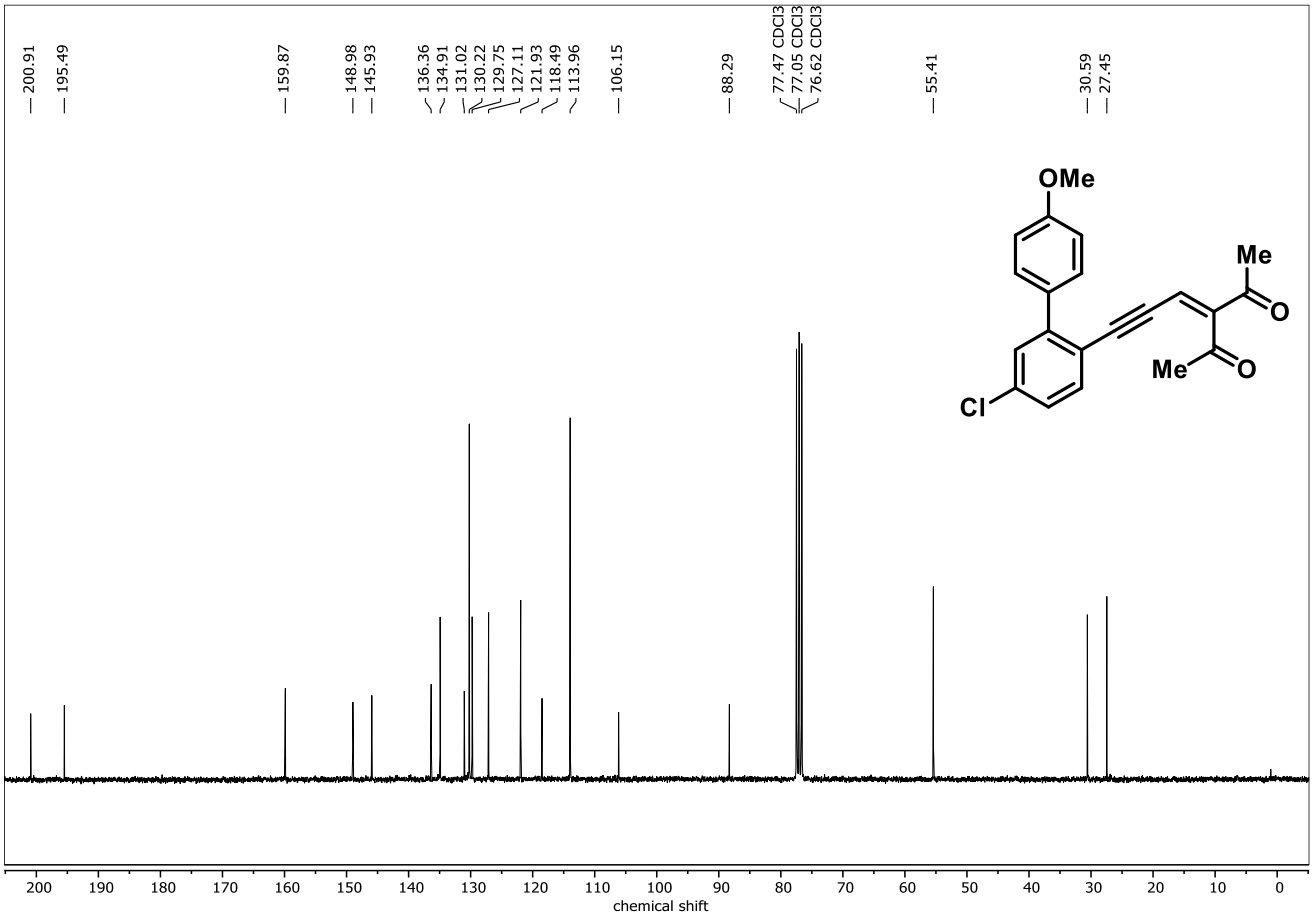
$^{13}C\{^{1}H\}$ (75 MHz, $CDCl_3$) NMR Spectrum of 2j



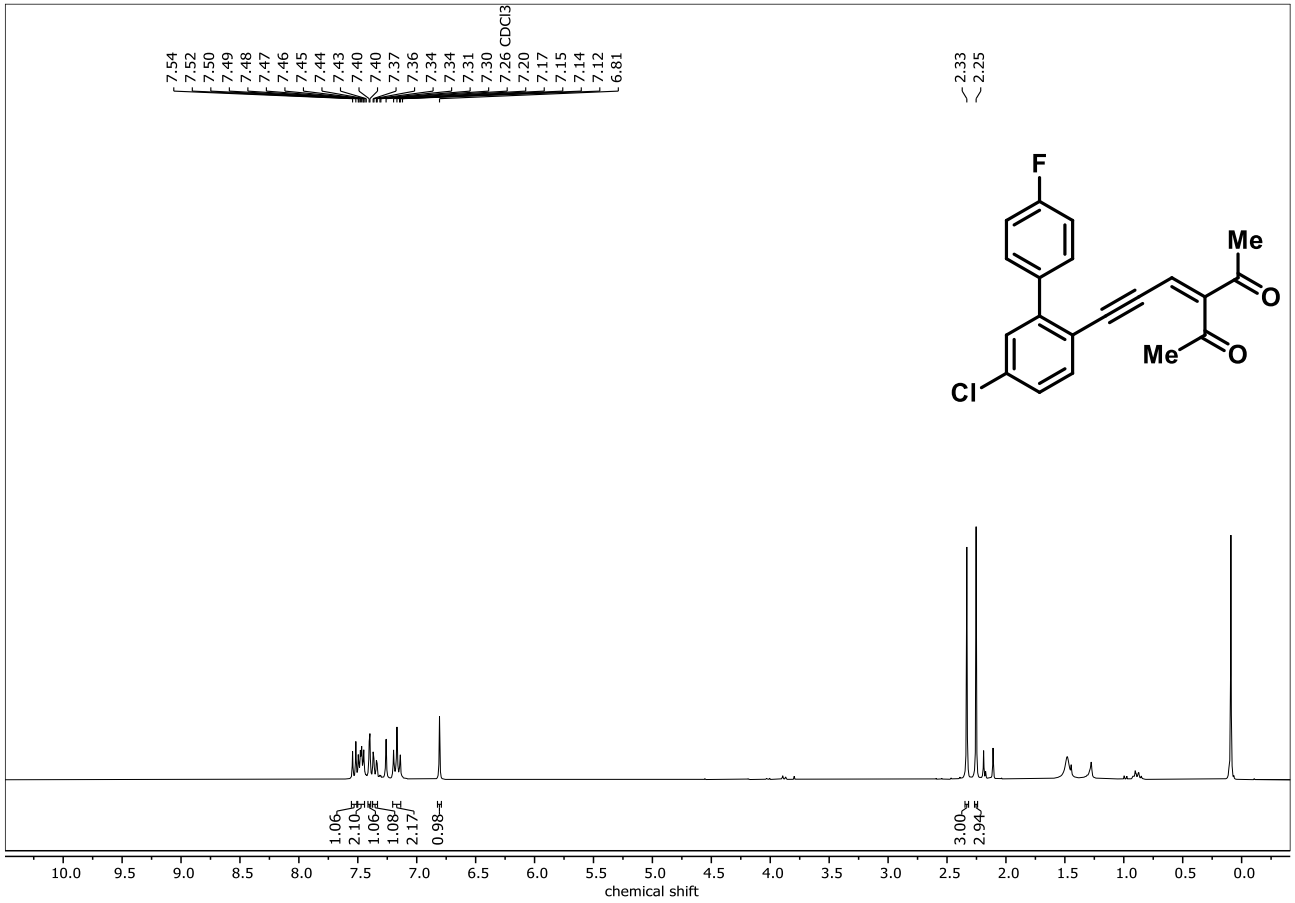
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 2k



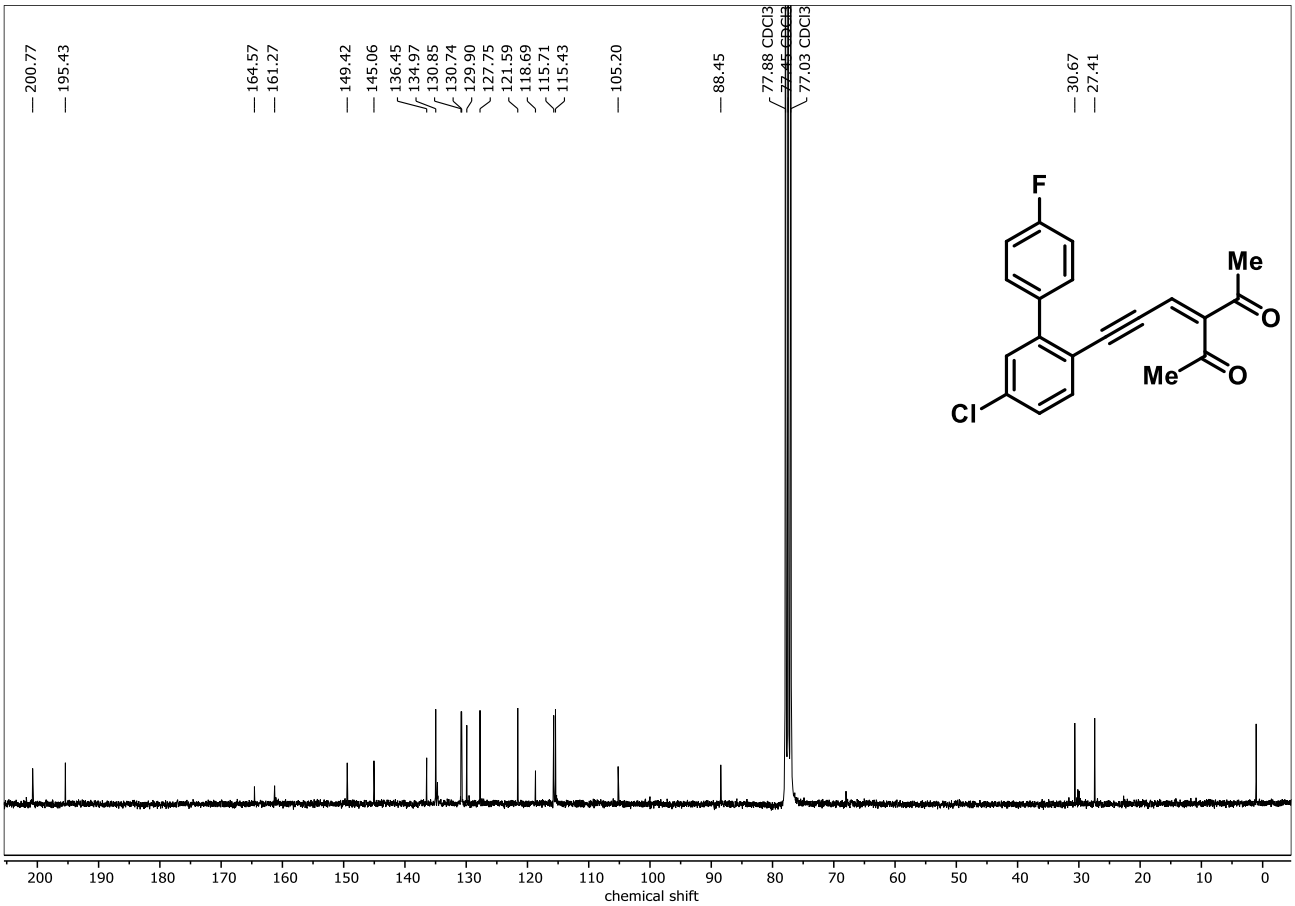
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2k



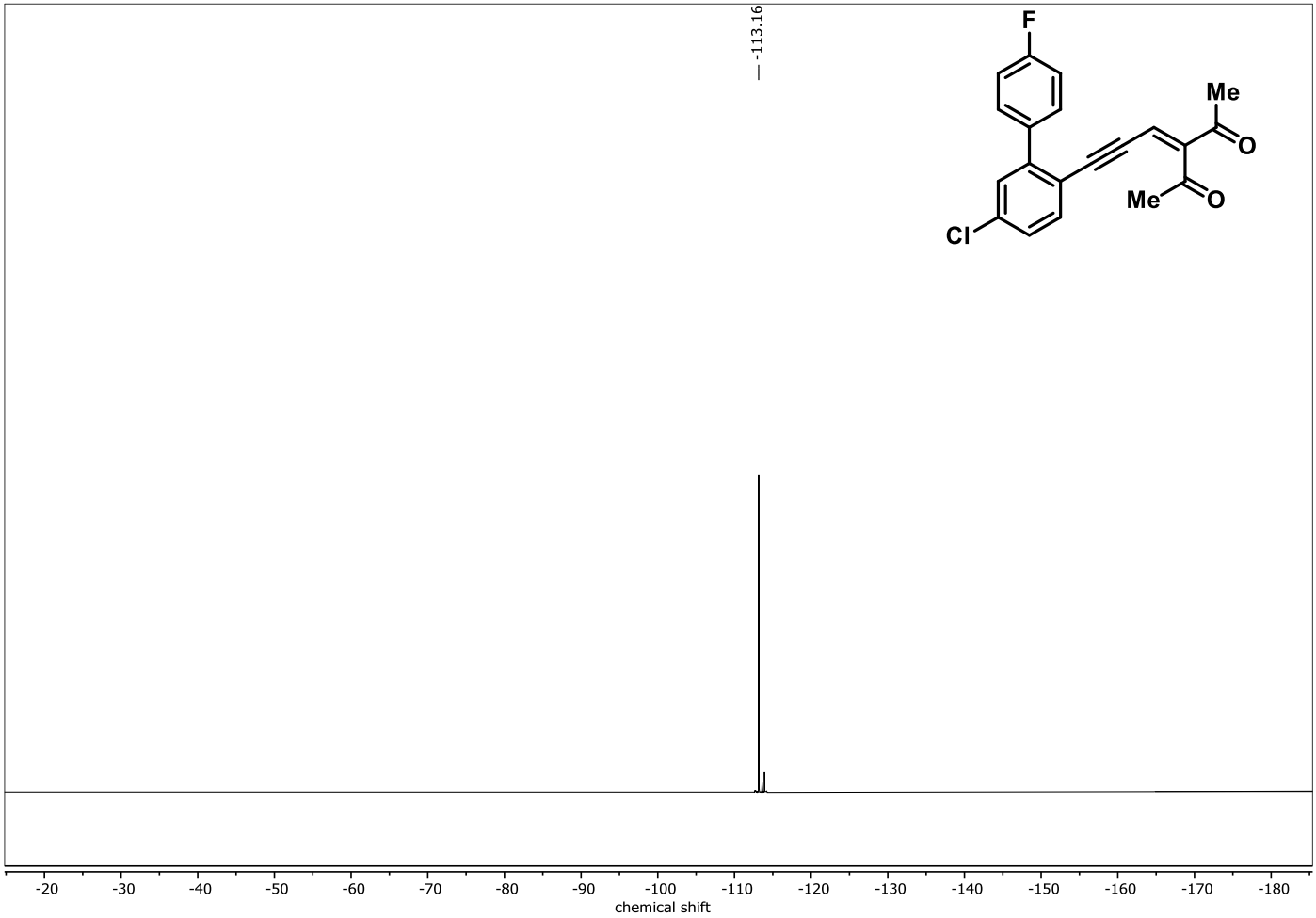
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2l



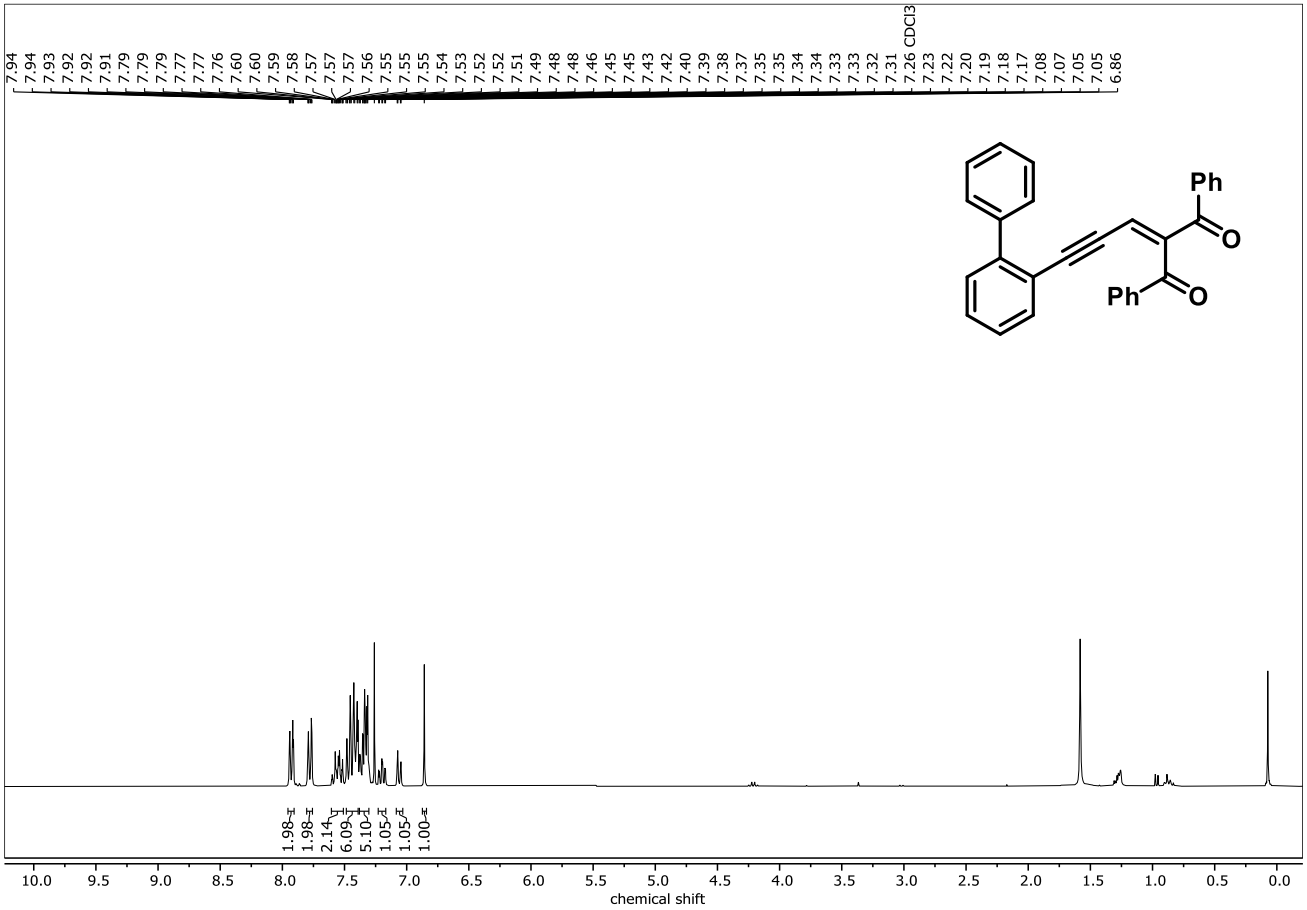
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2l



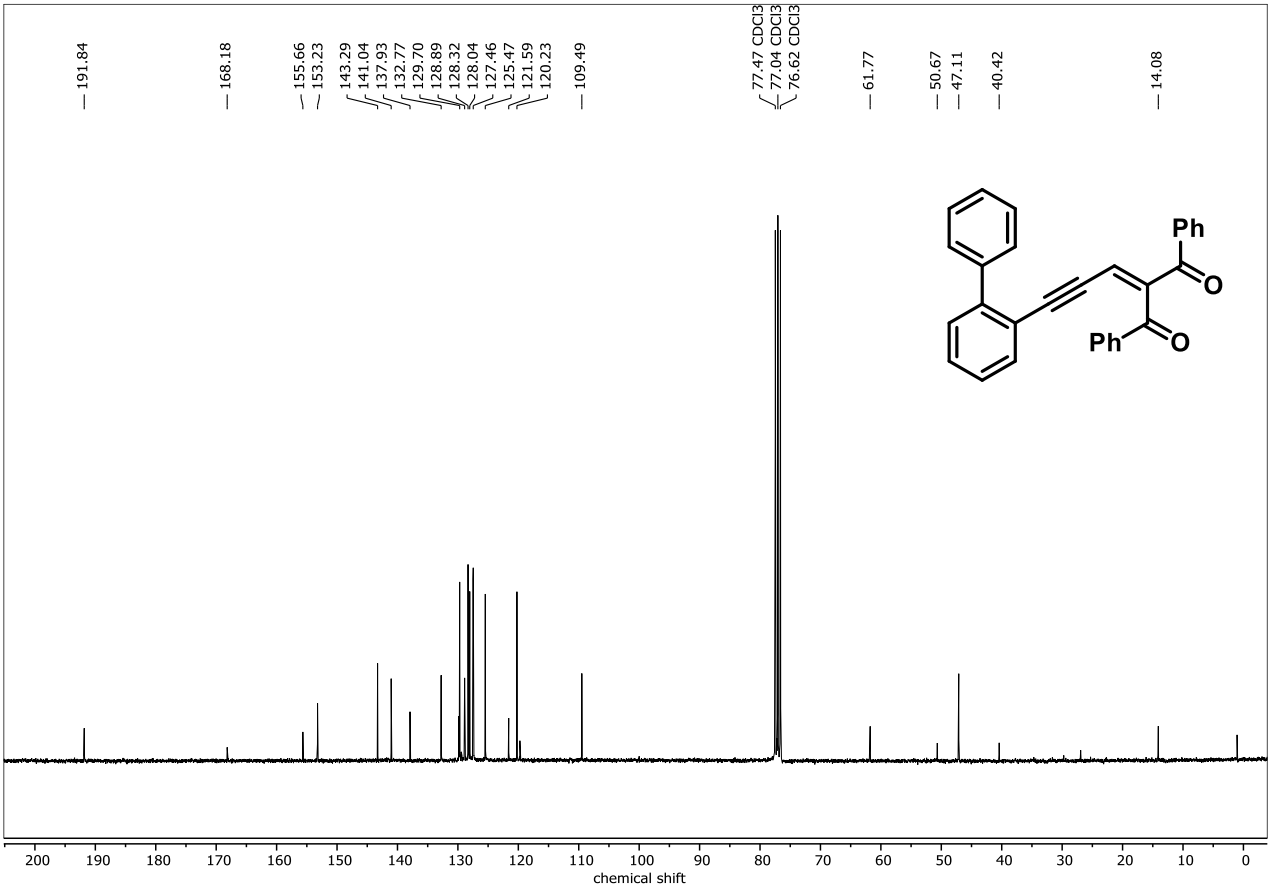
^{19}F (282 MHz, $CDCl_3$) NMR Spectrum of 2l



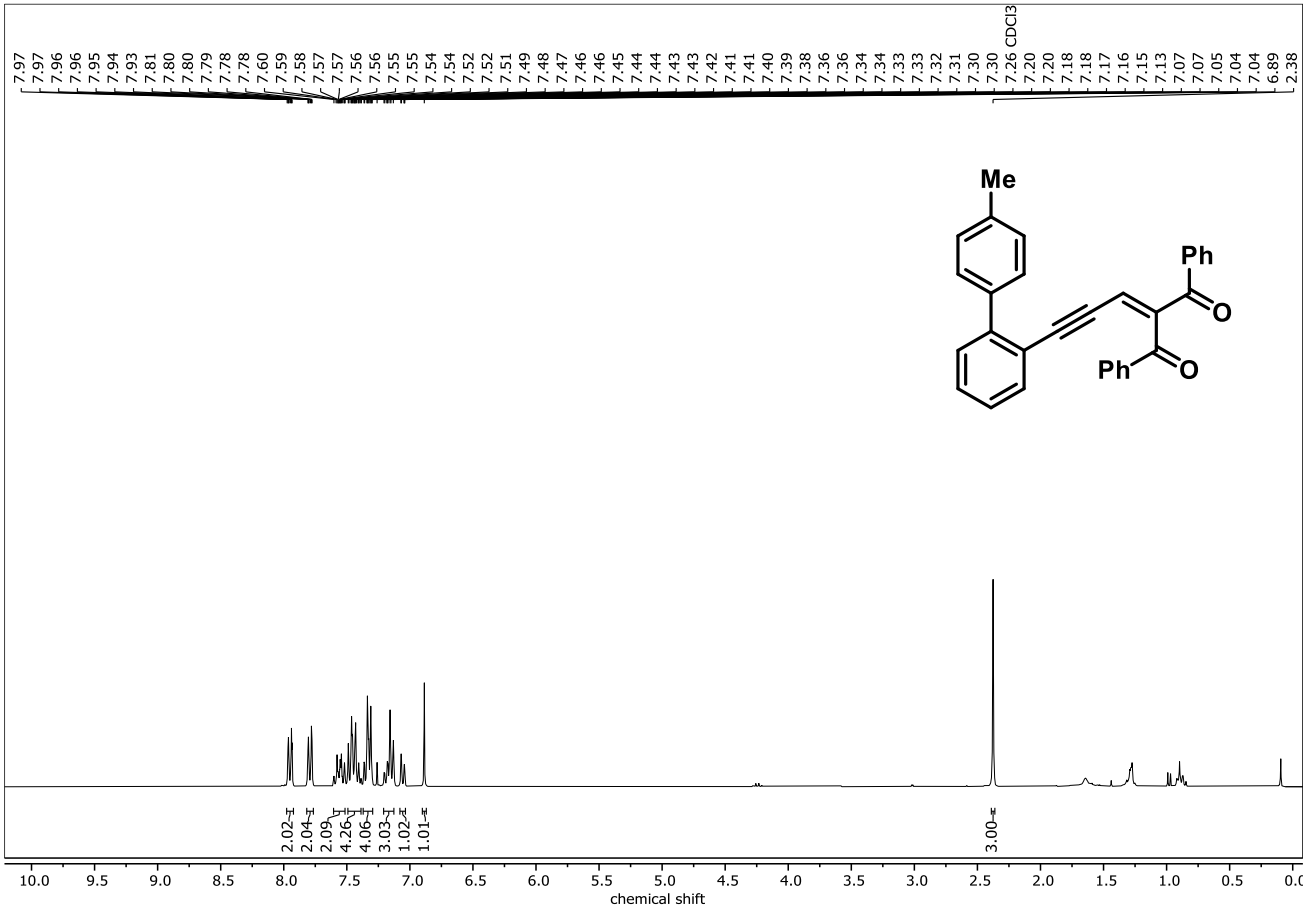
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2m



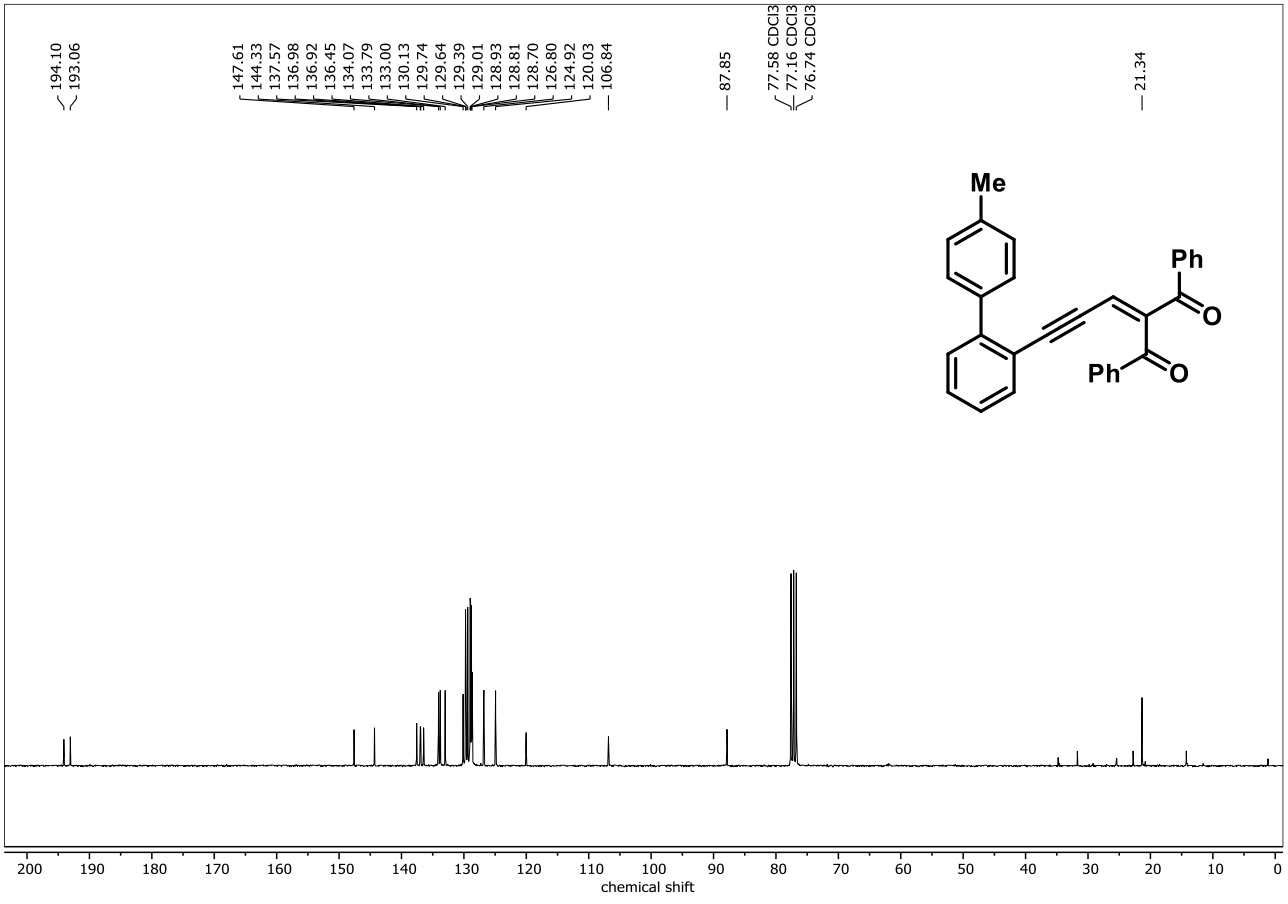
$^{13}C\{^{1}H\}$ (75 MHz, $CDCl_3$) NMR Spectrum of 2m



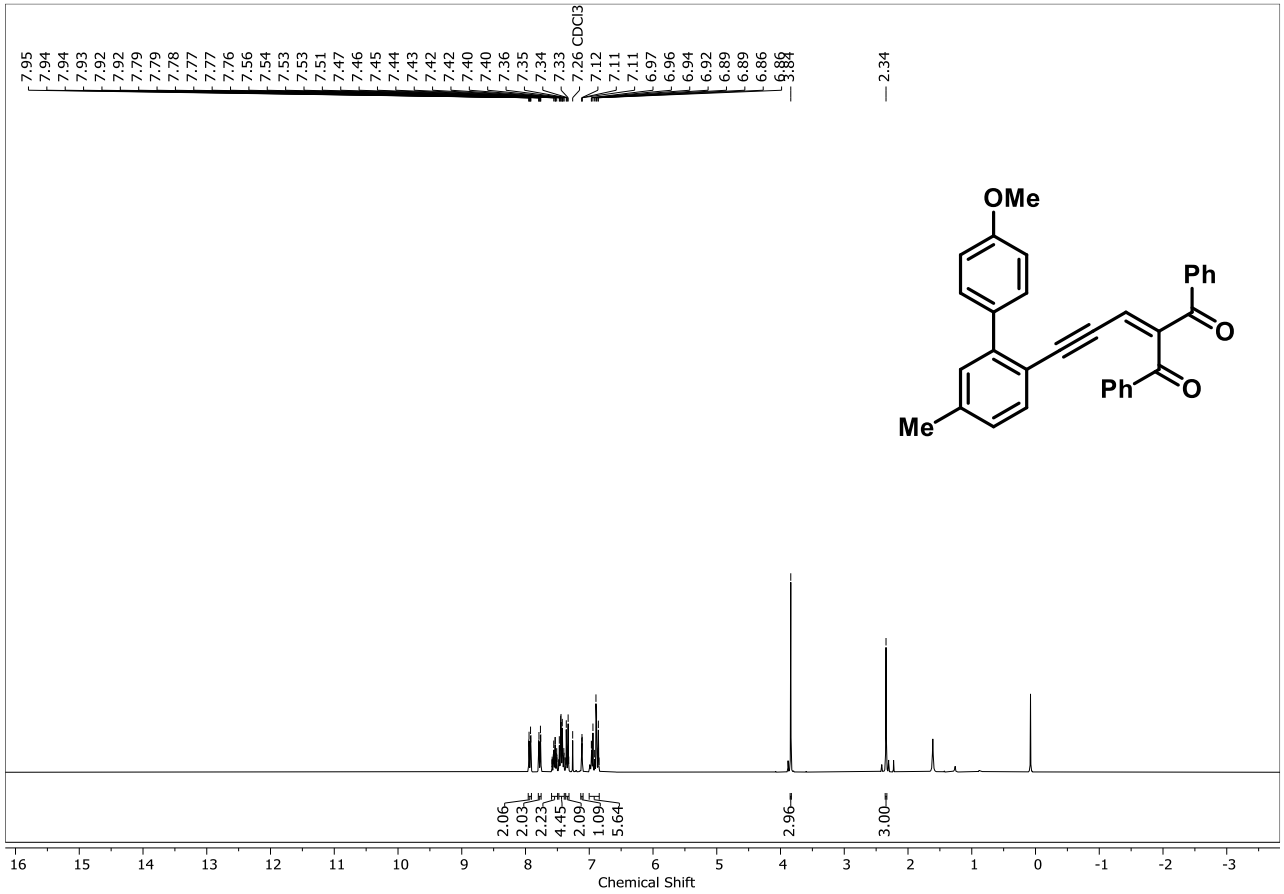
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2n



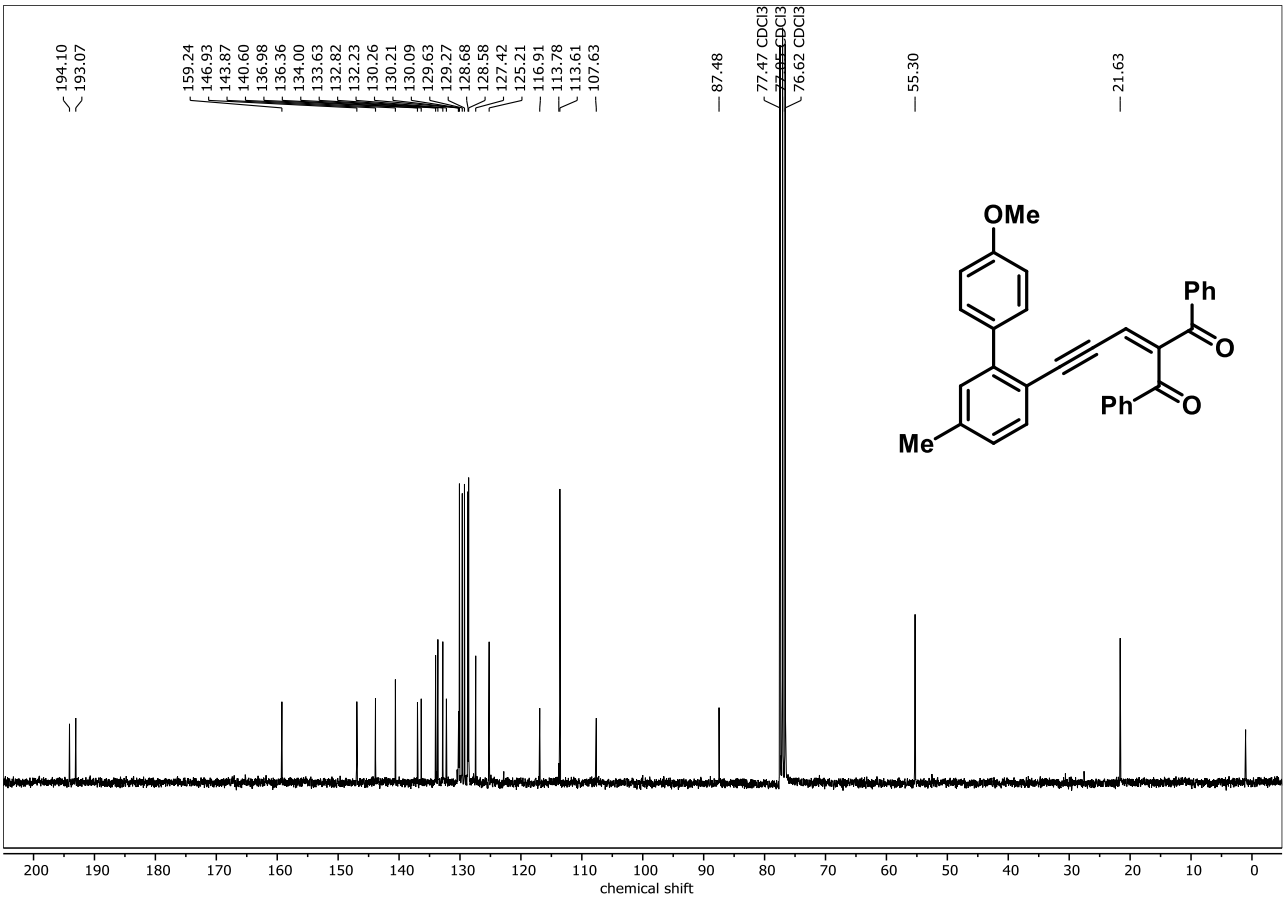
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2n



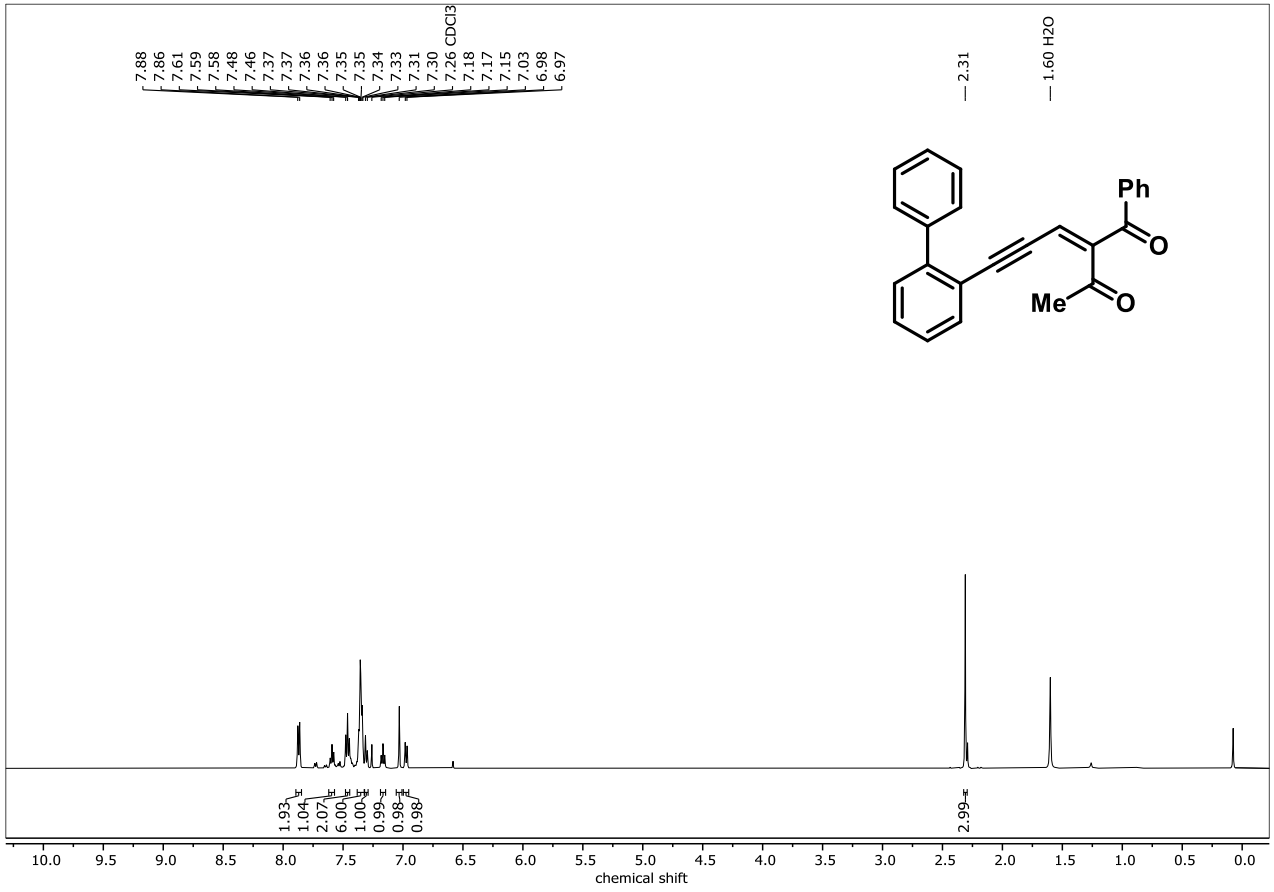
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 2o



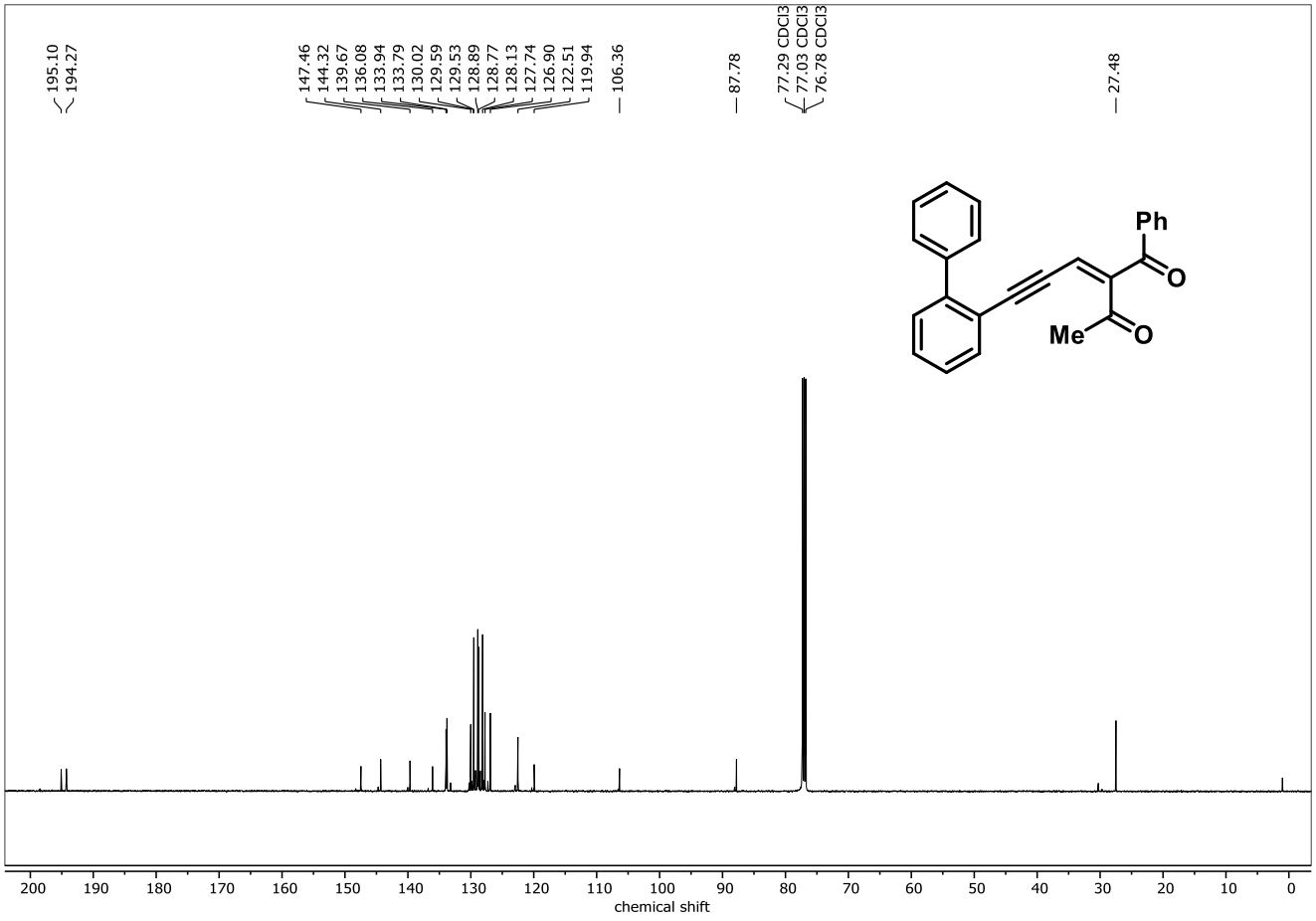
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 2o



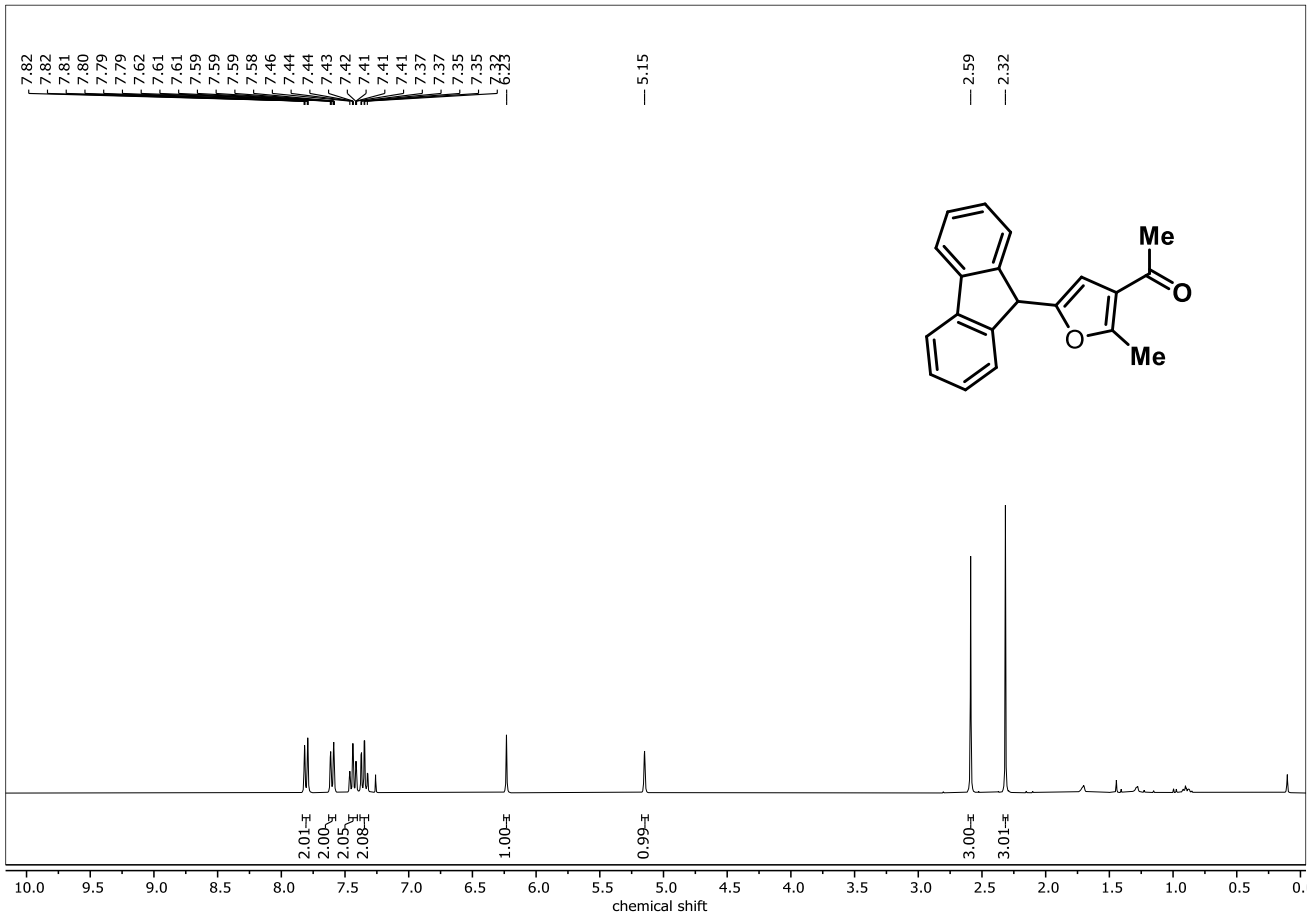
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 2p



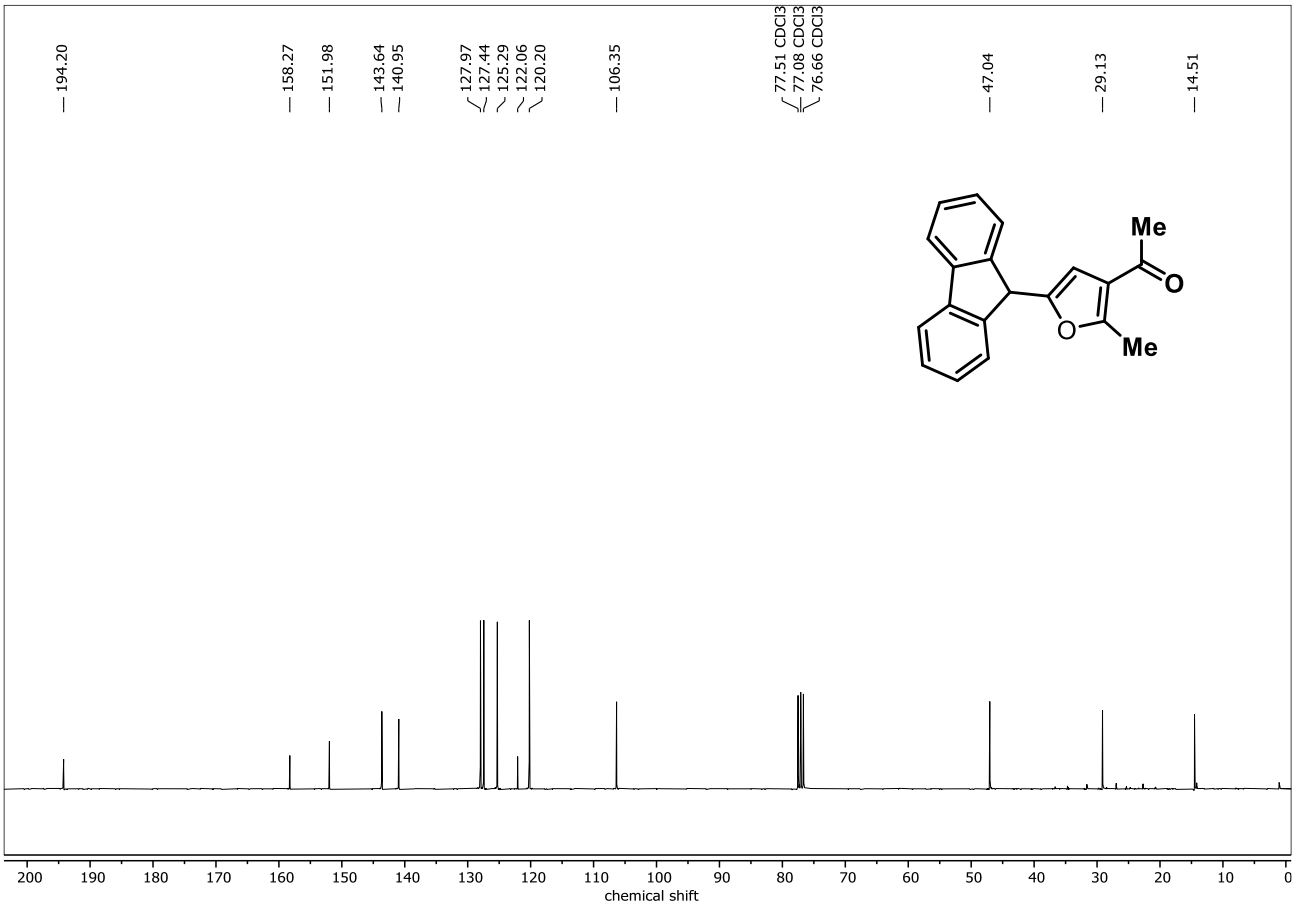
^{13}C{^{1}H} (126 MHz, $CDCl_3$) NMR Spectrum of 2p



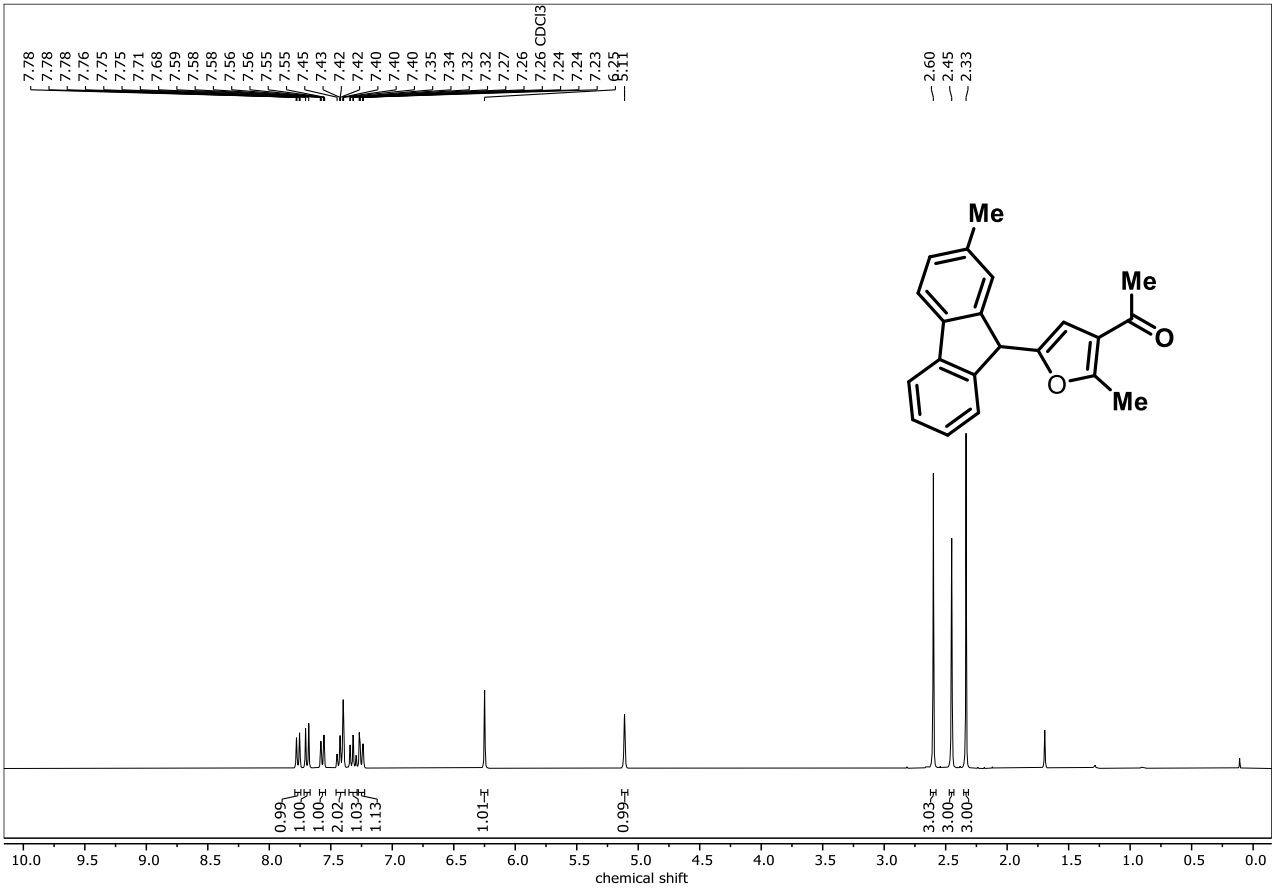
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3a



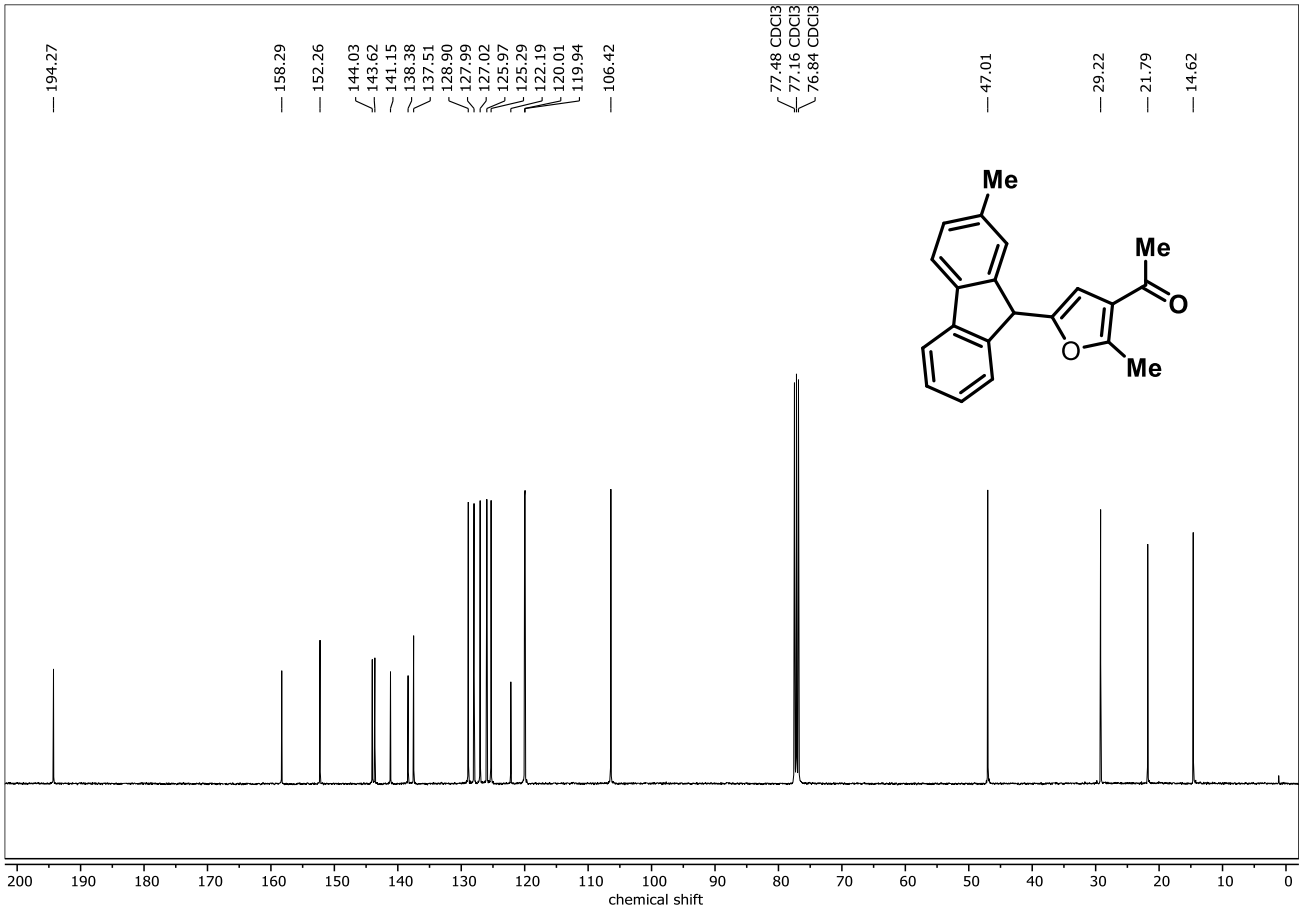
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3a



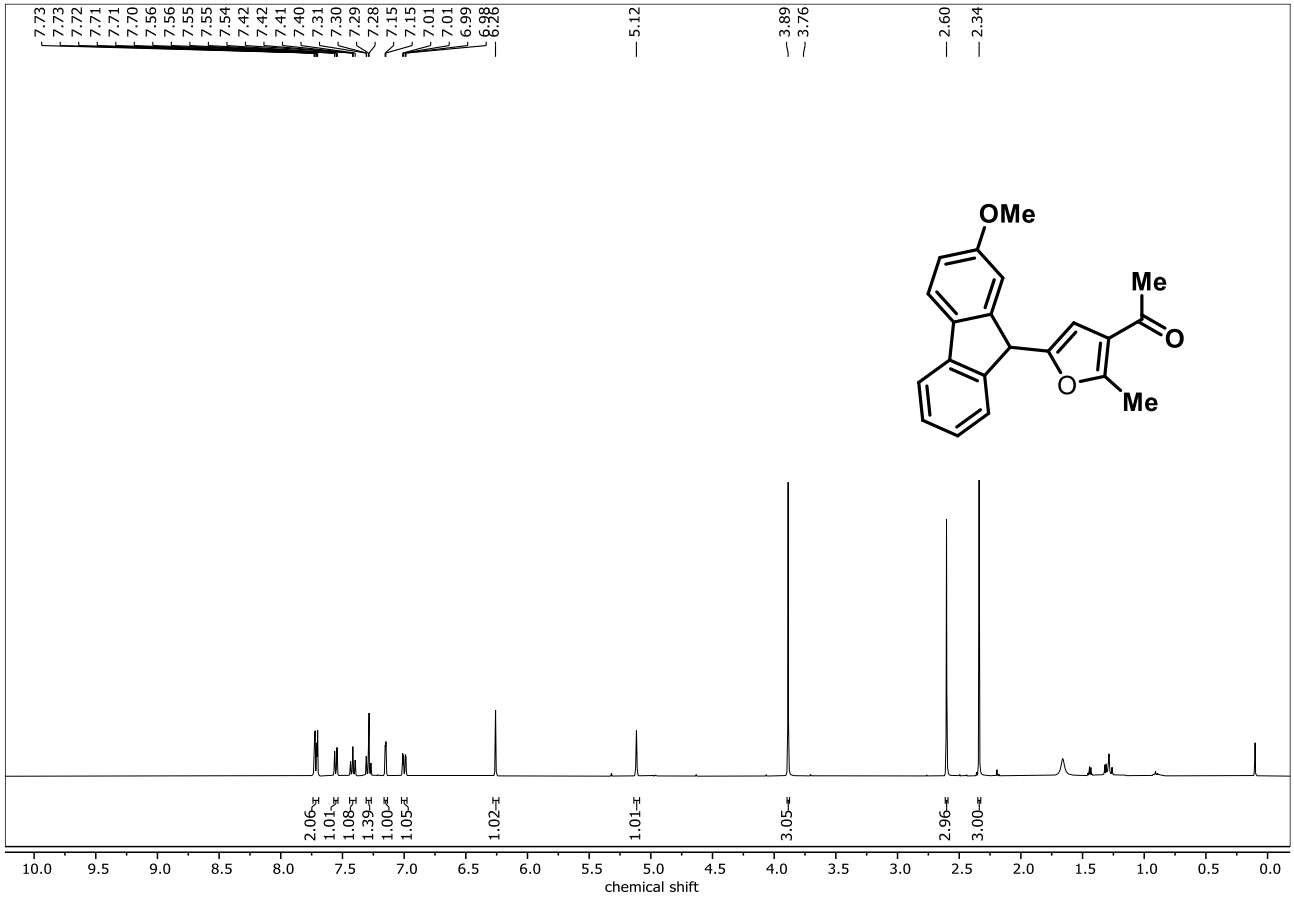
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3b



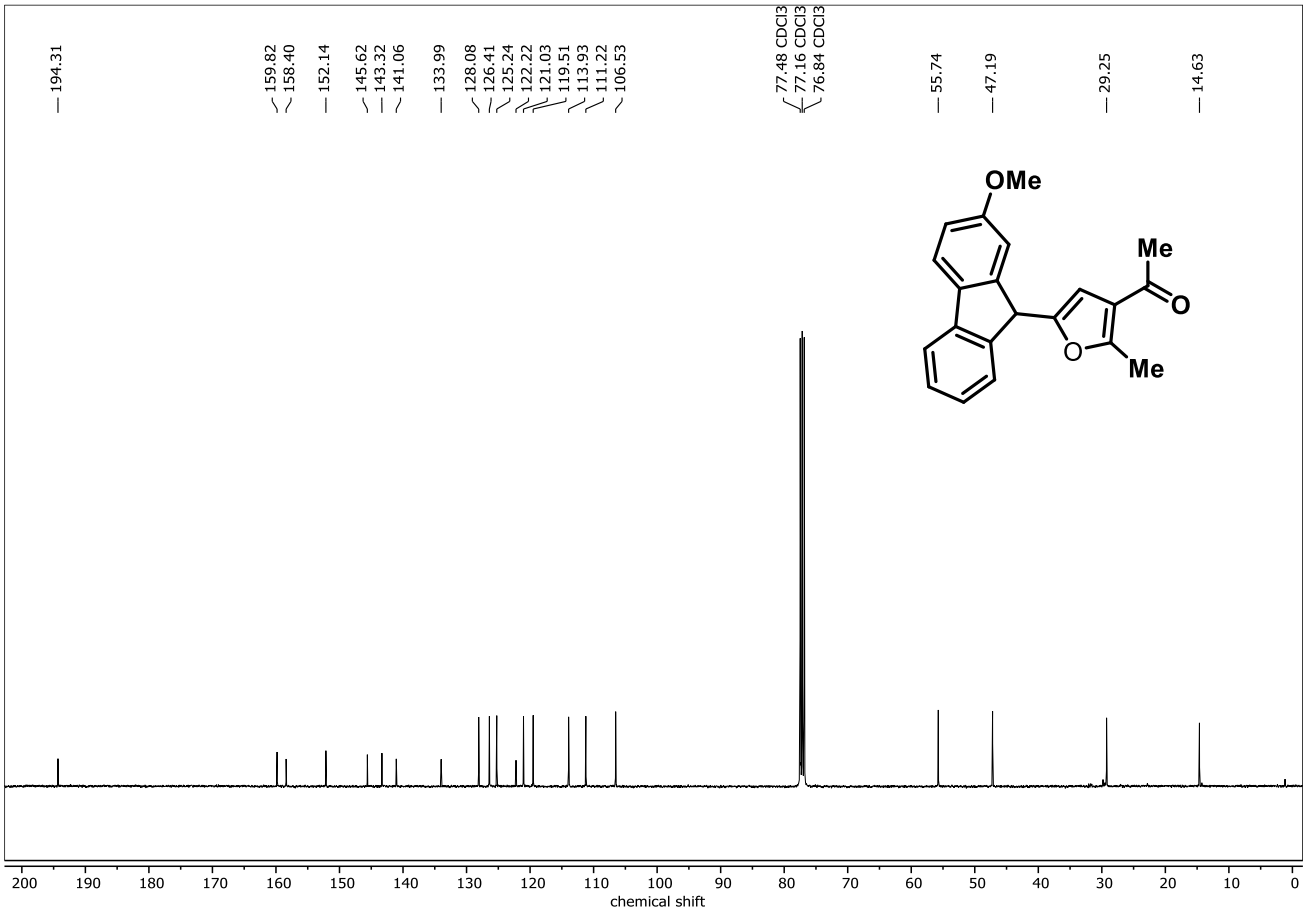
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3b



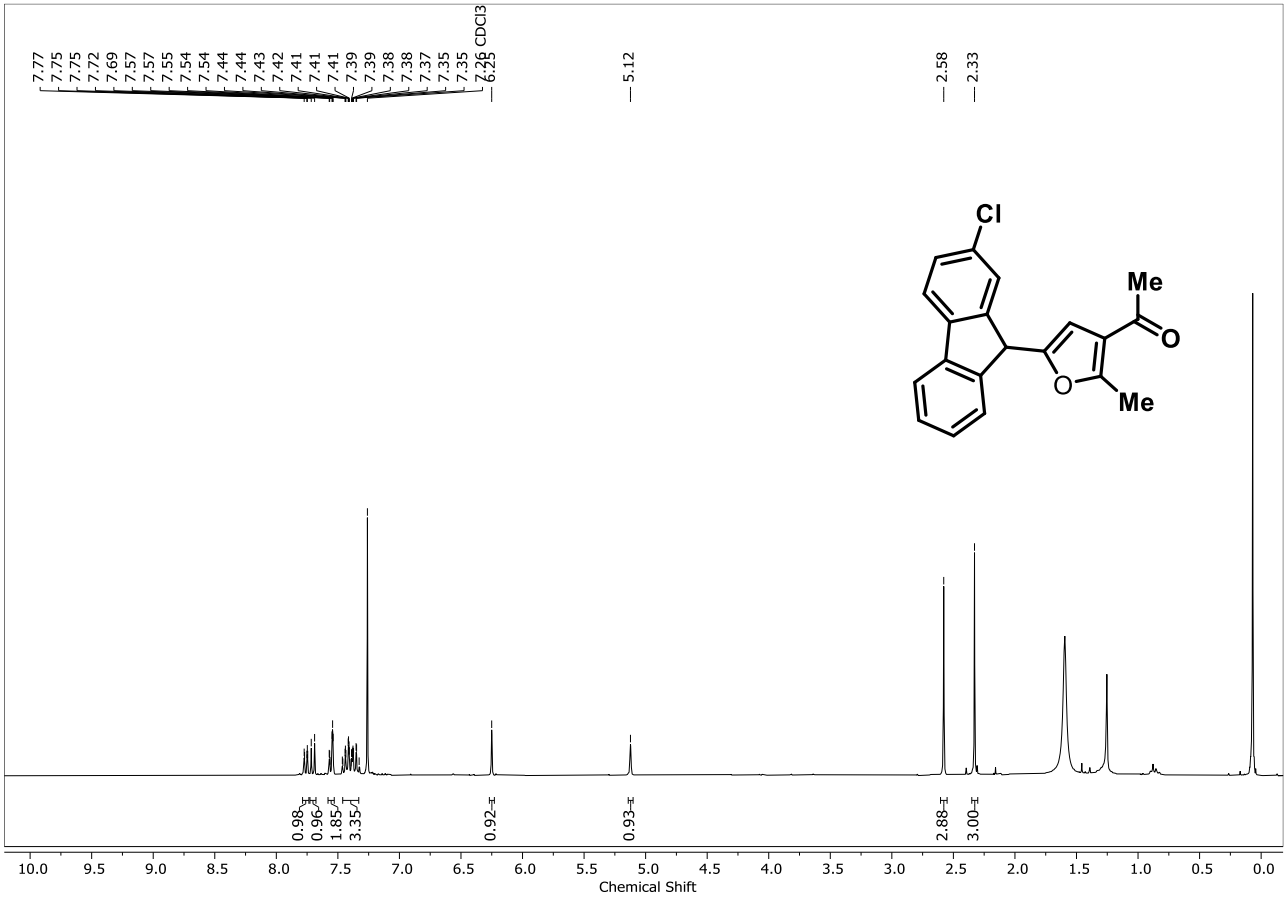
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 3c



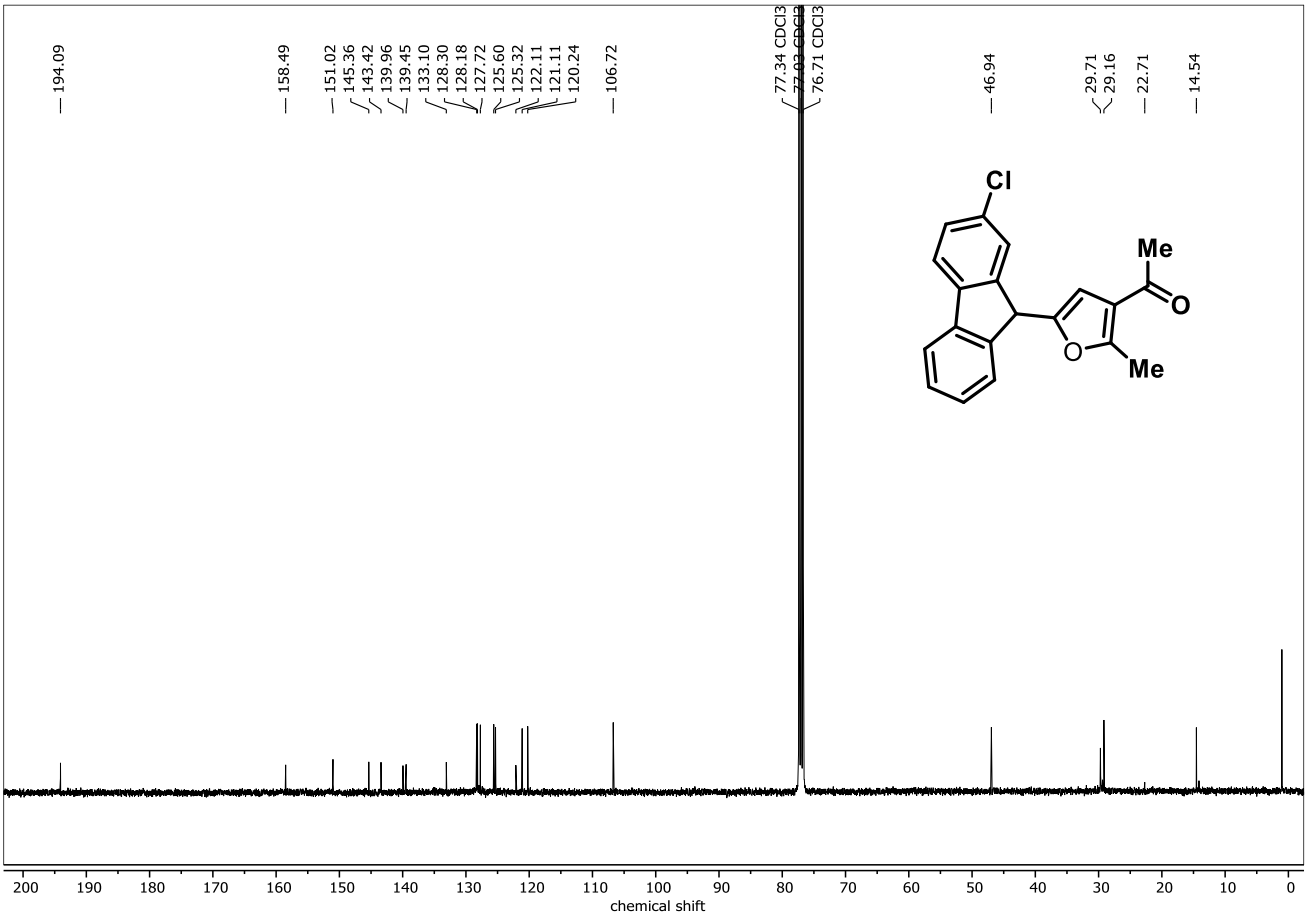
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3c



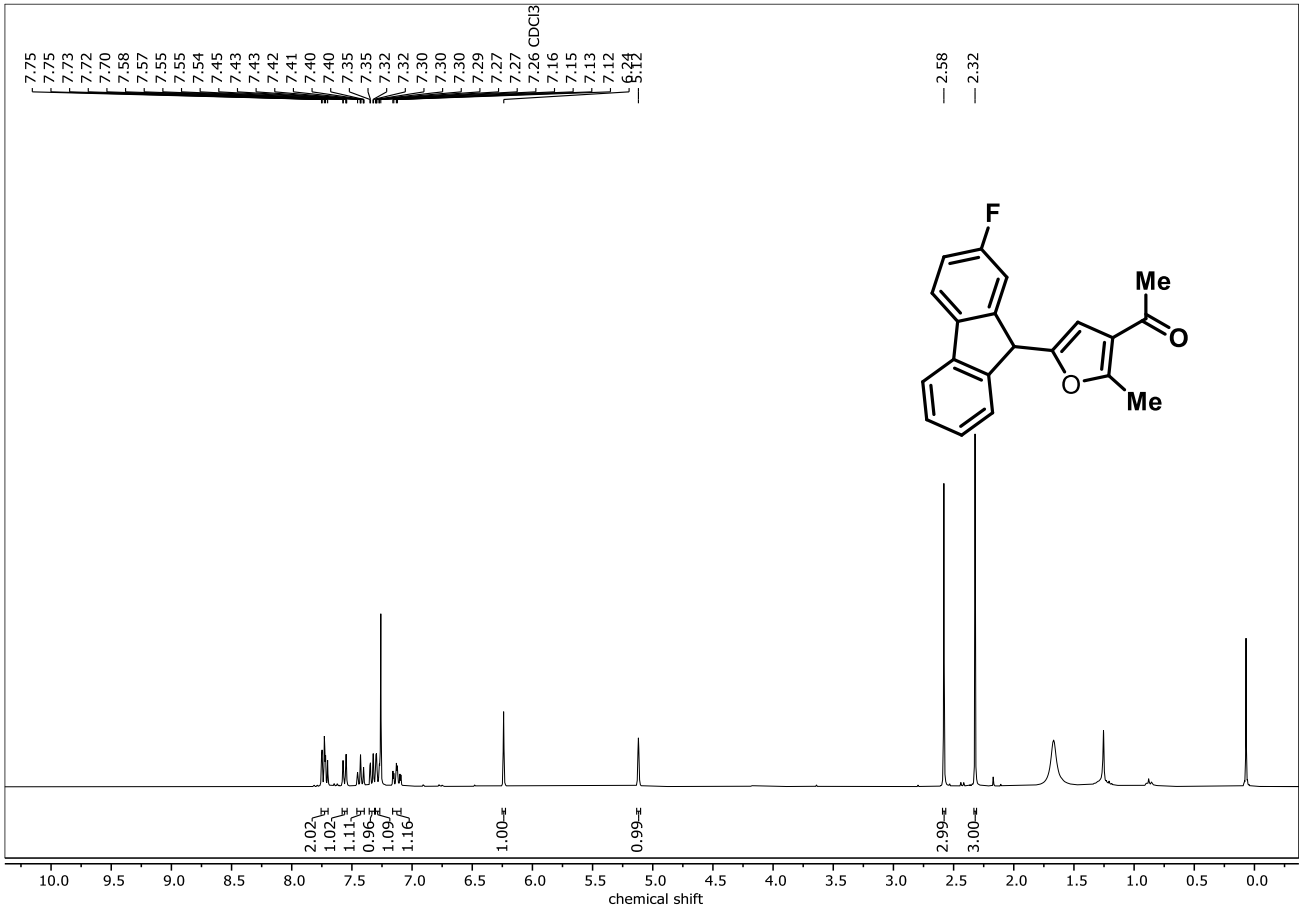
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3d



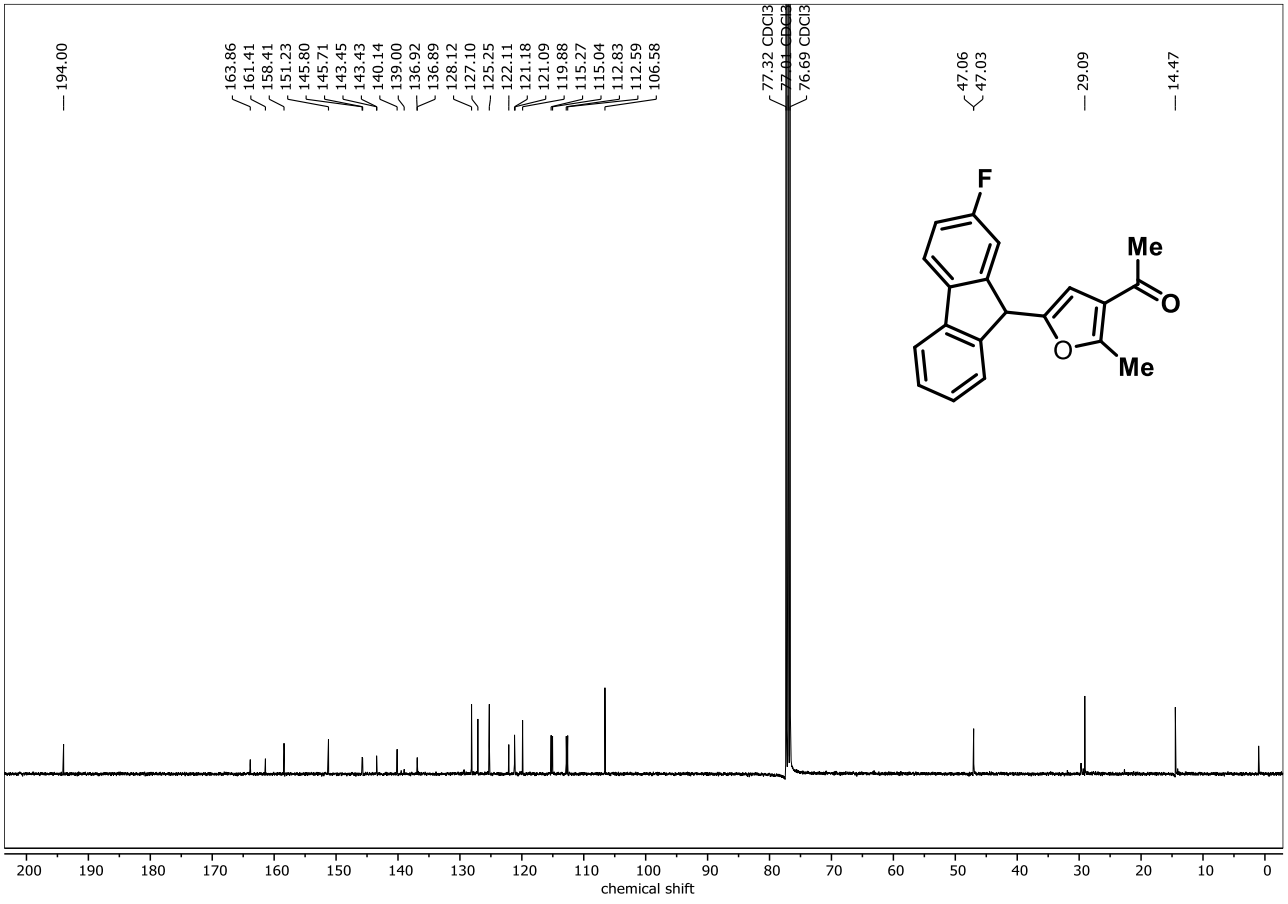
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3d



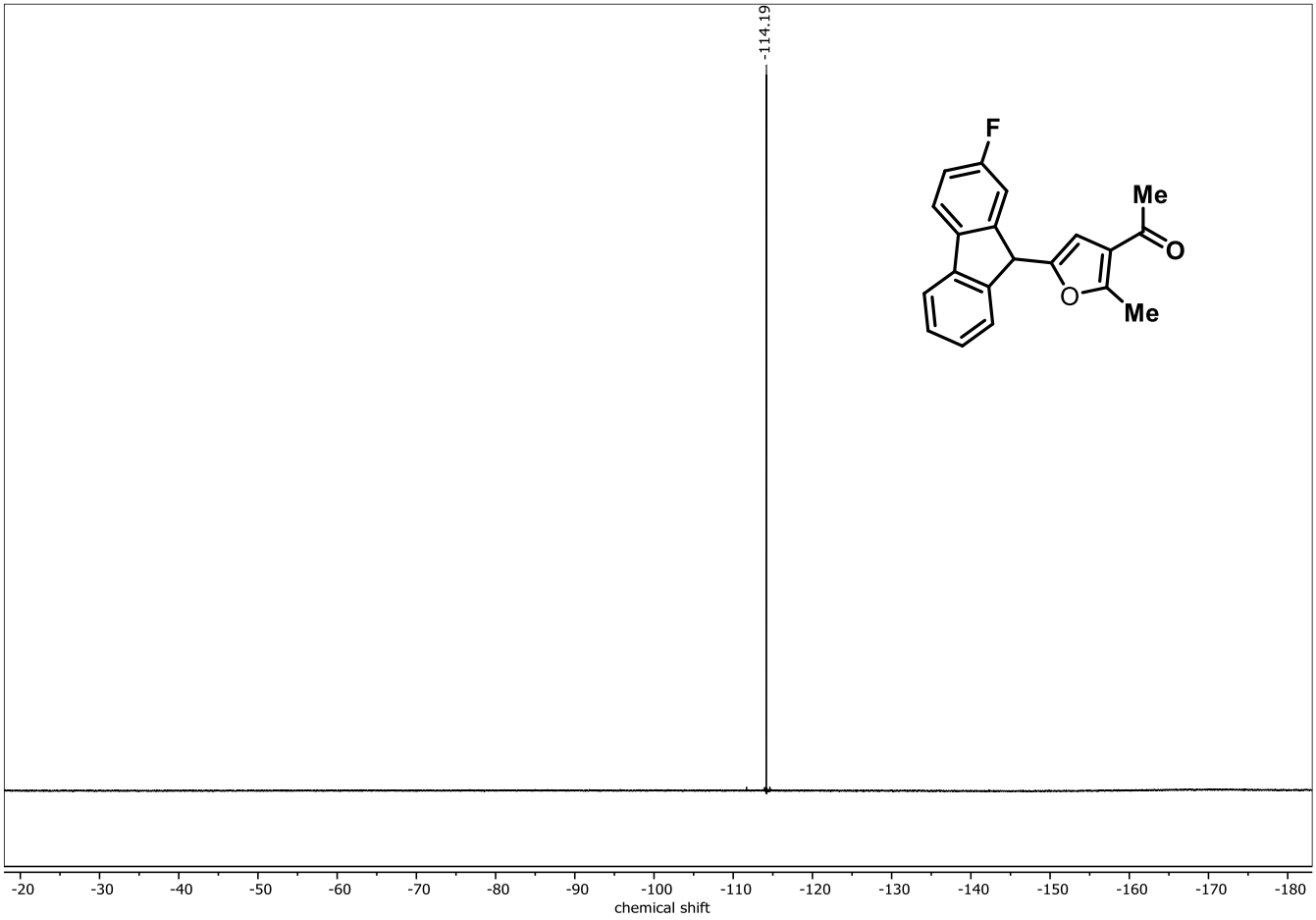
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3e



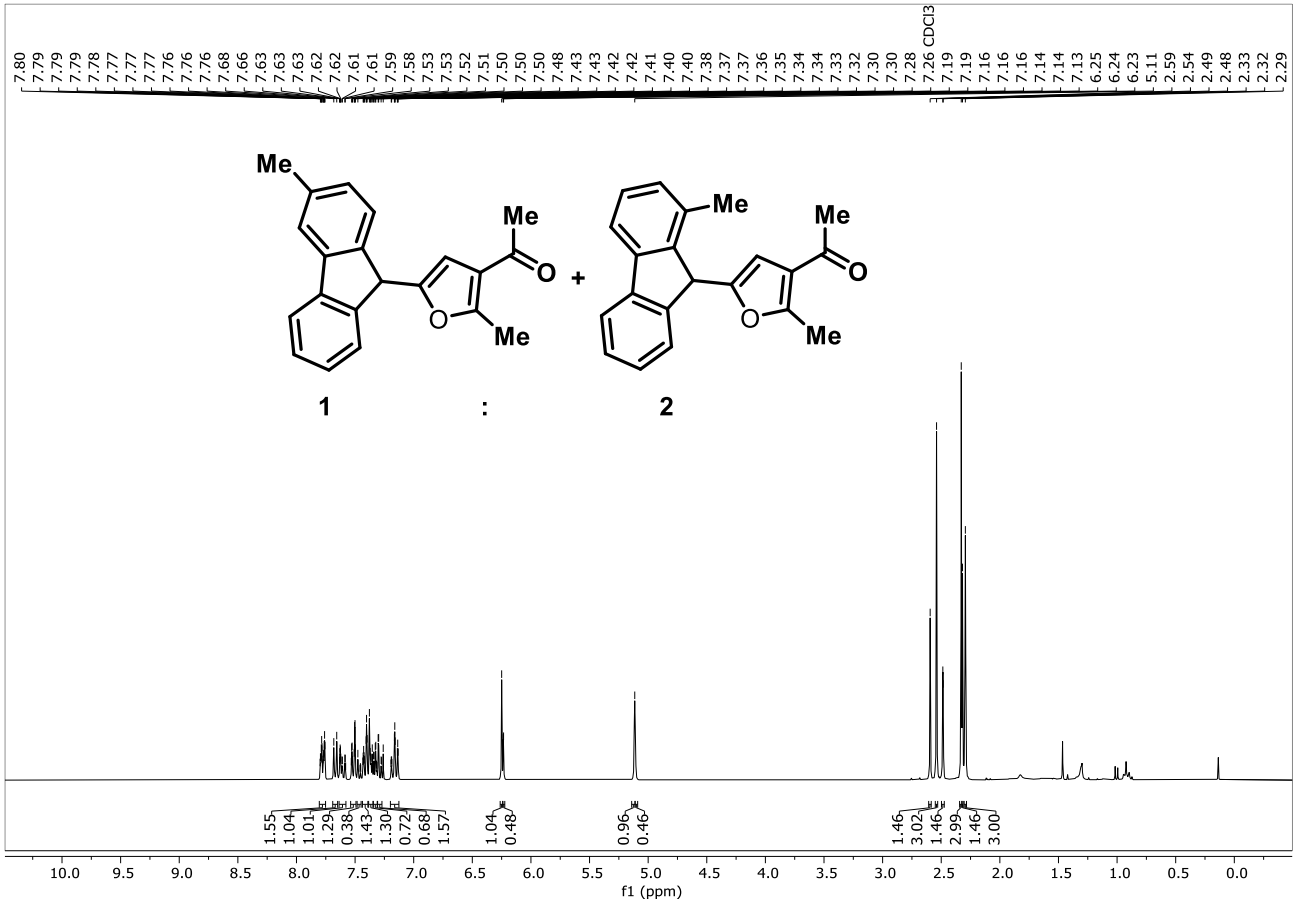
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3e



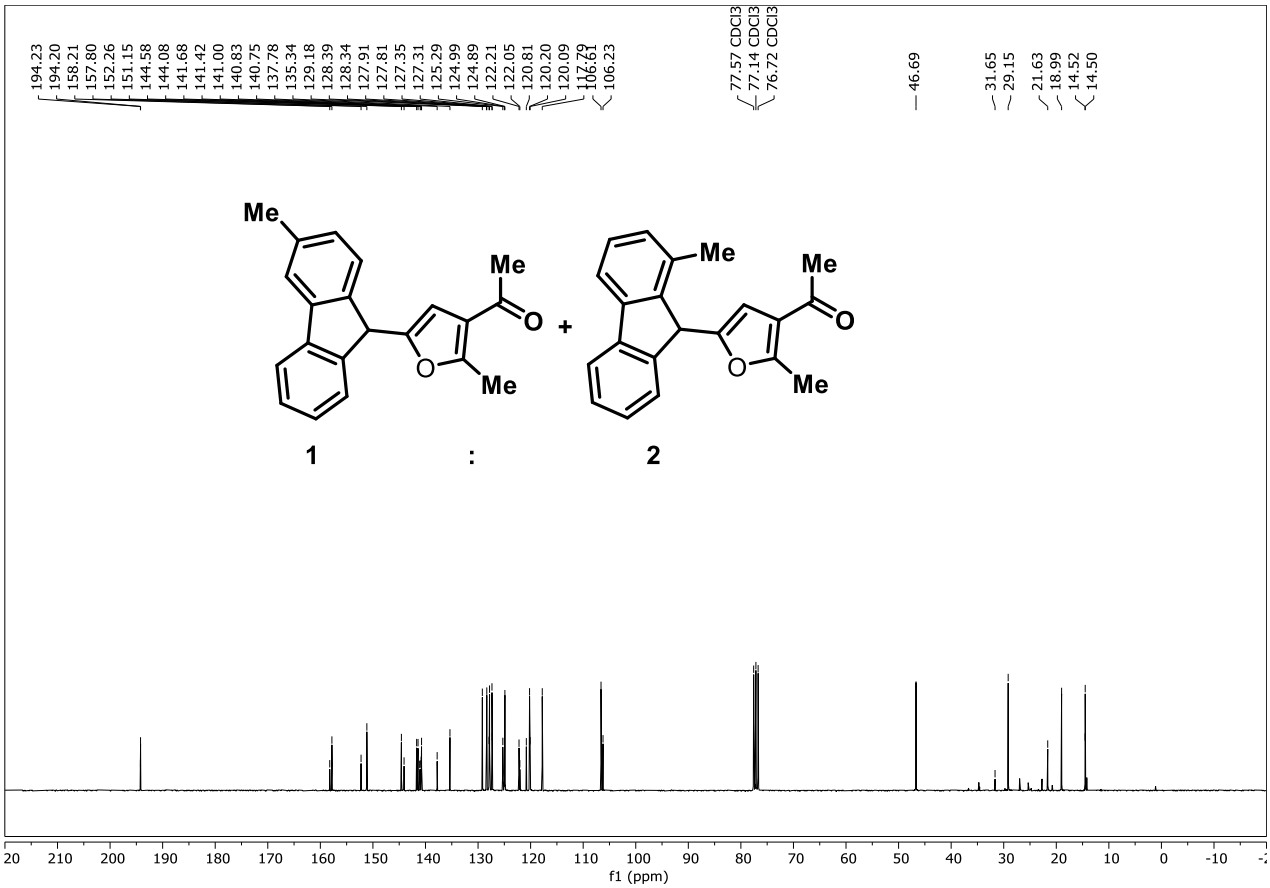
^{19}F NMR (377 MHz, $CDCl_3$) Spectrum of 3e



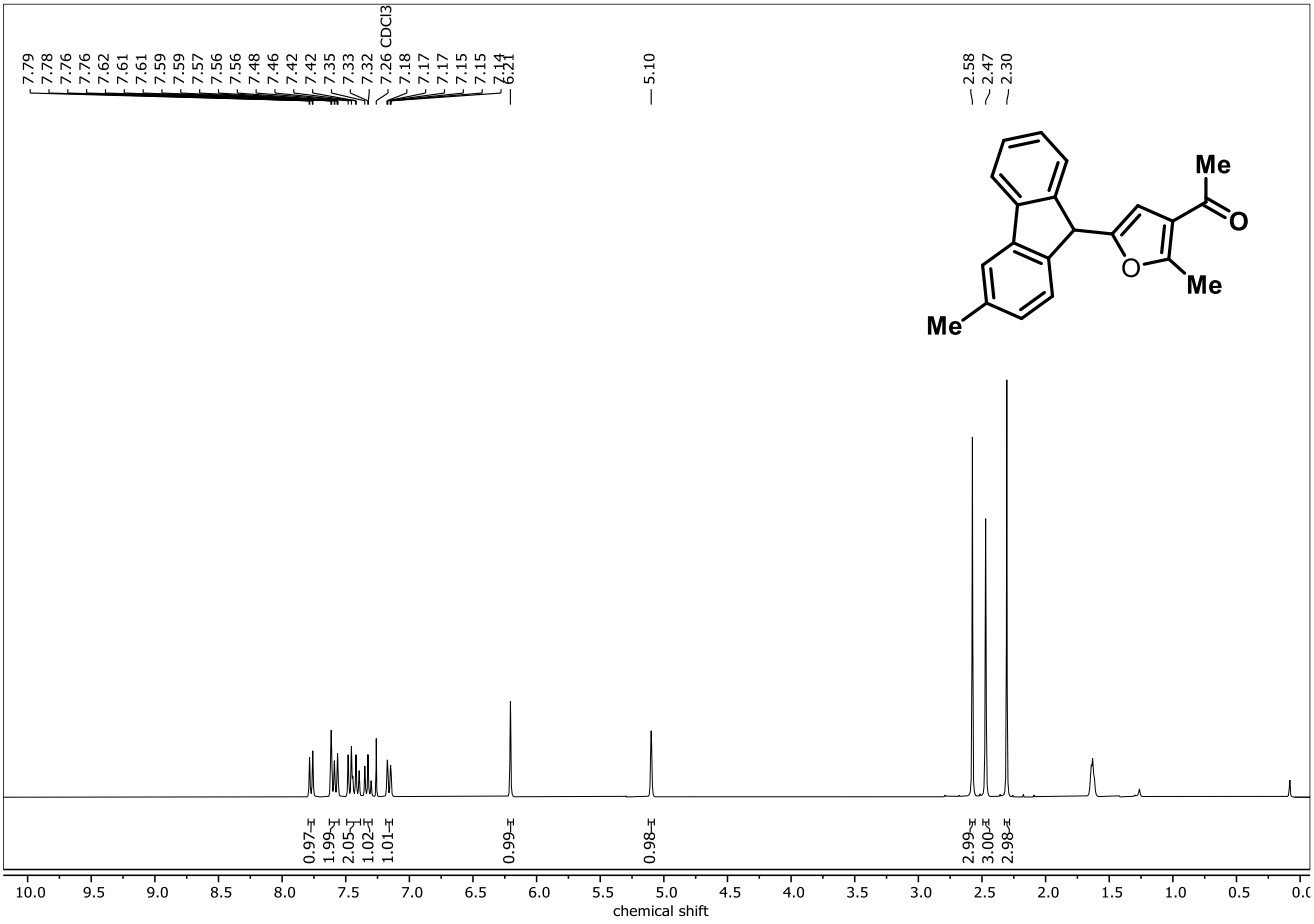
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3f



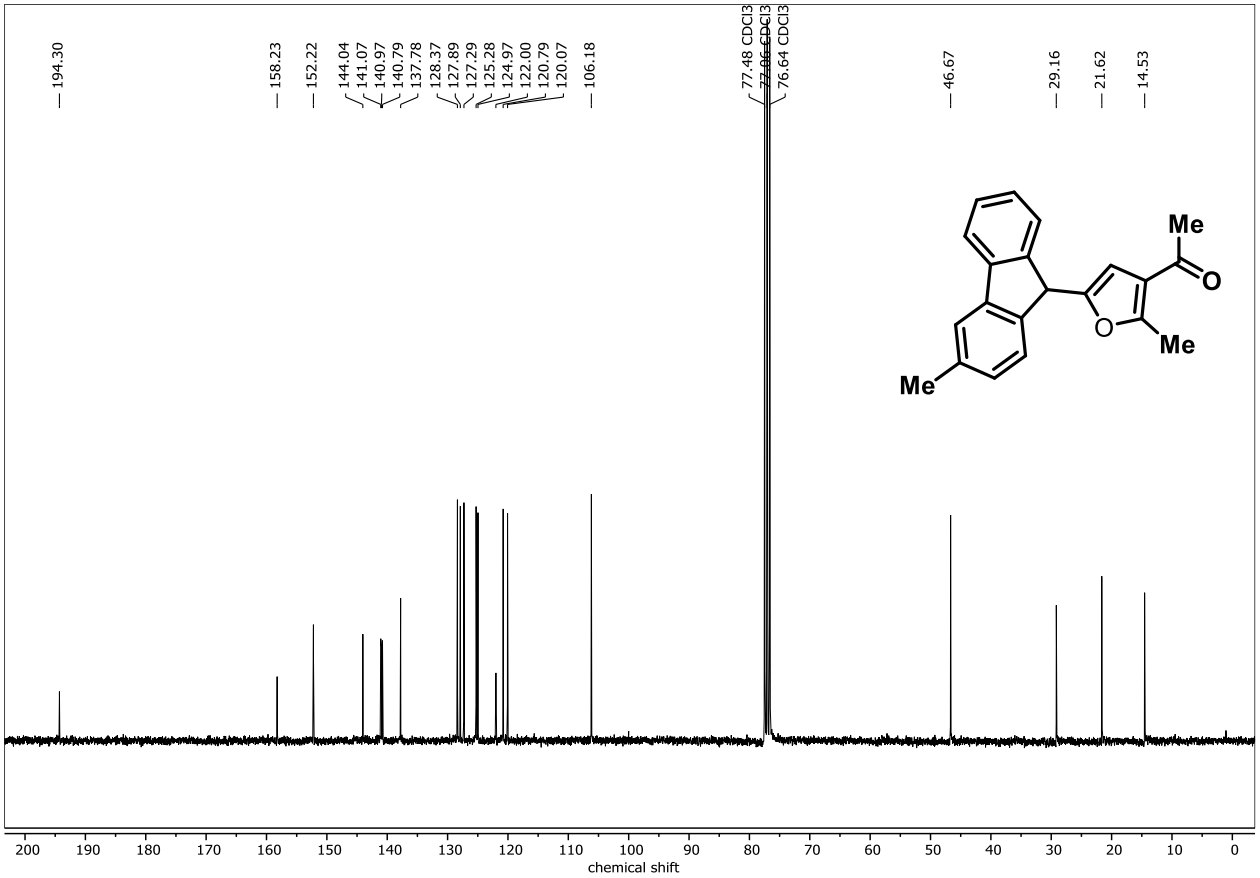
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3f



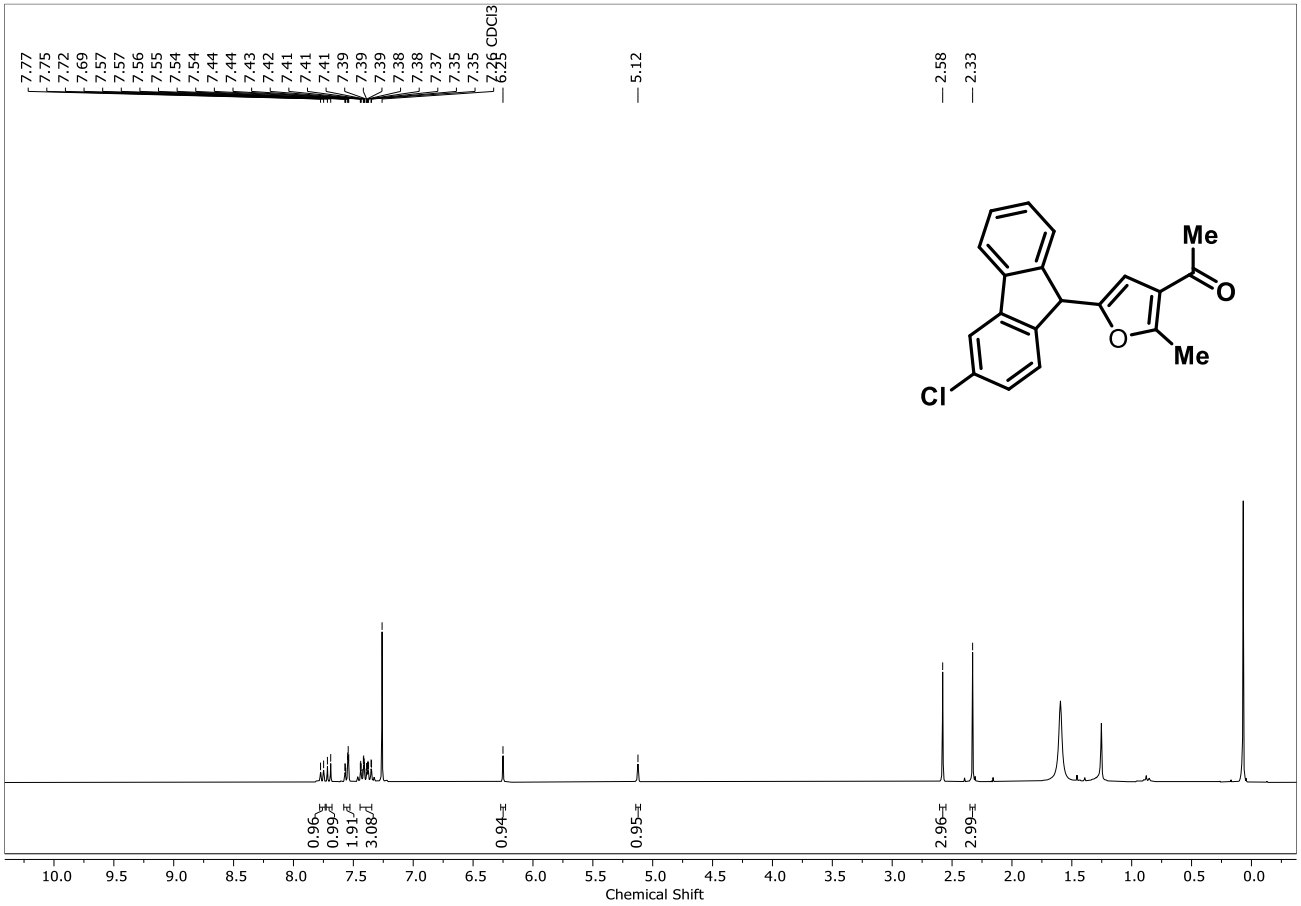
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3g



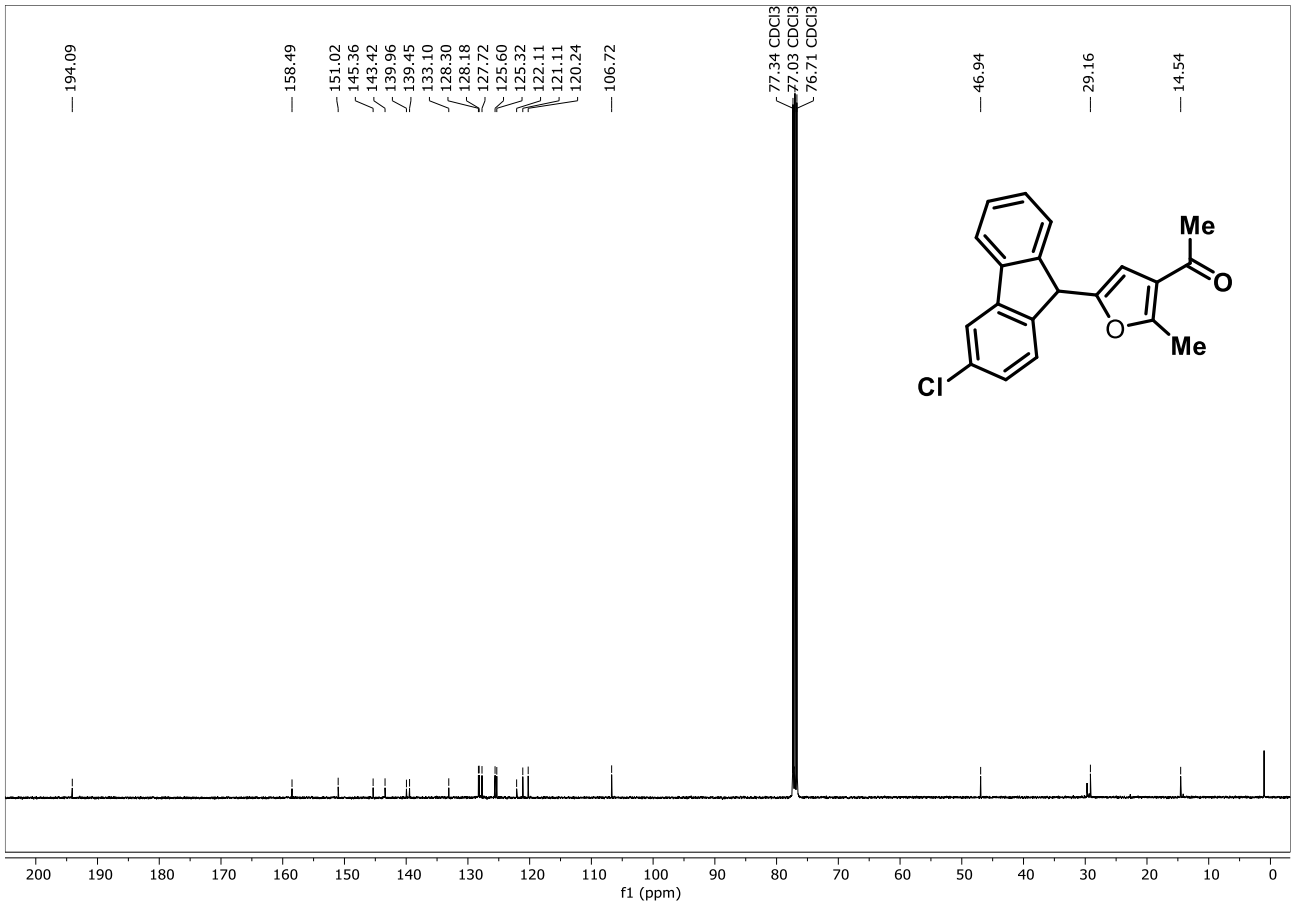
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3g



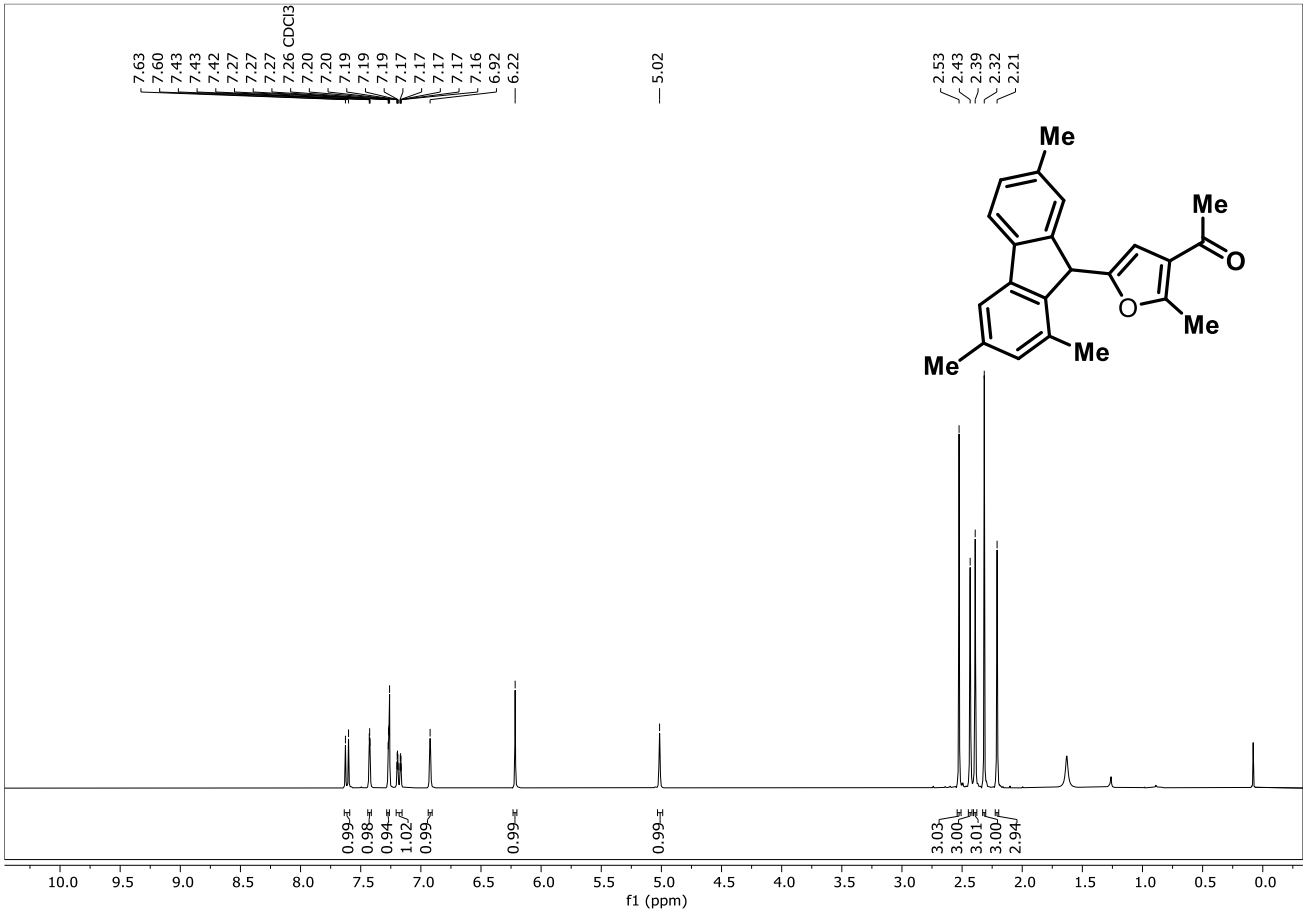
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3h



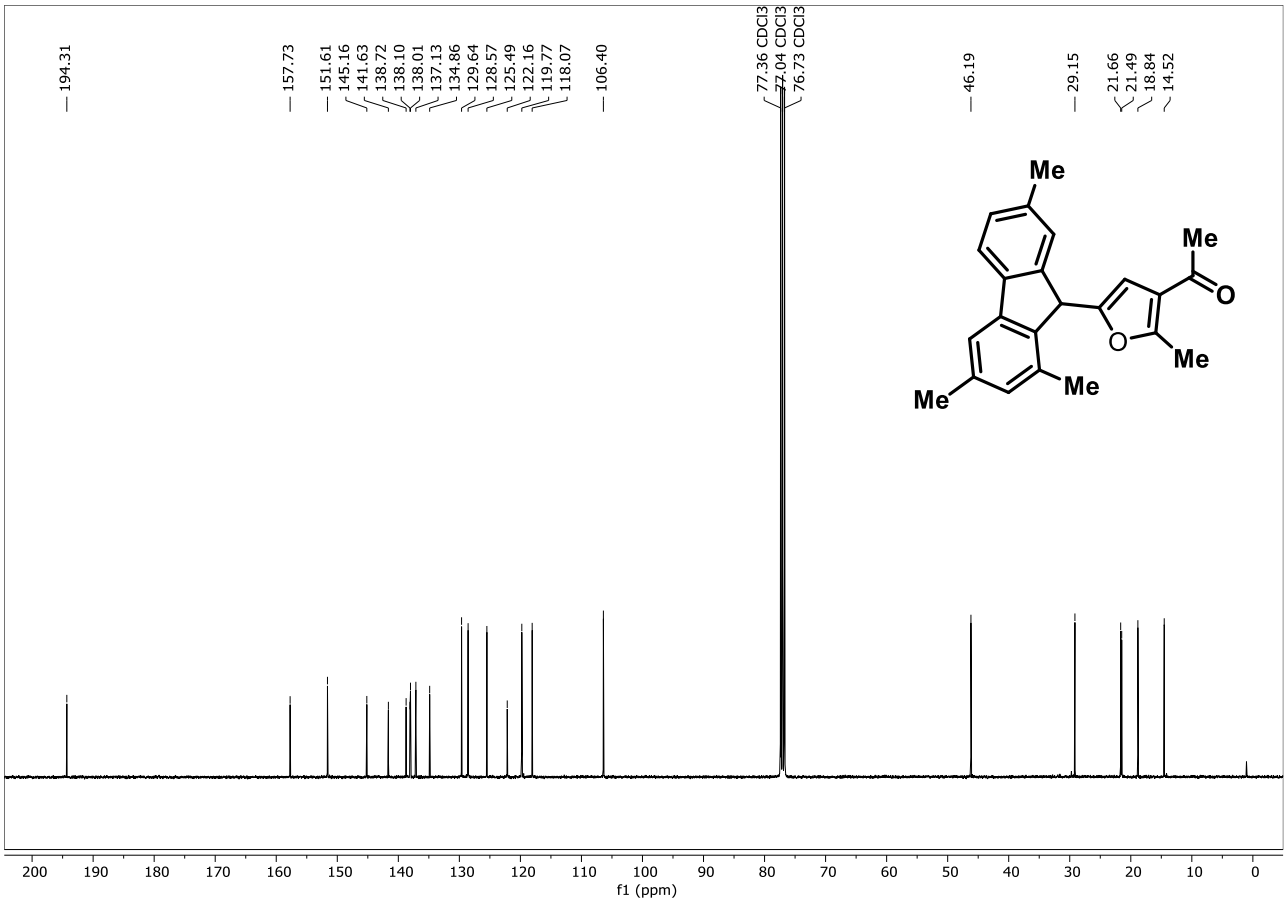
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3h



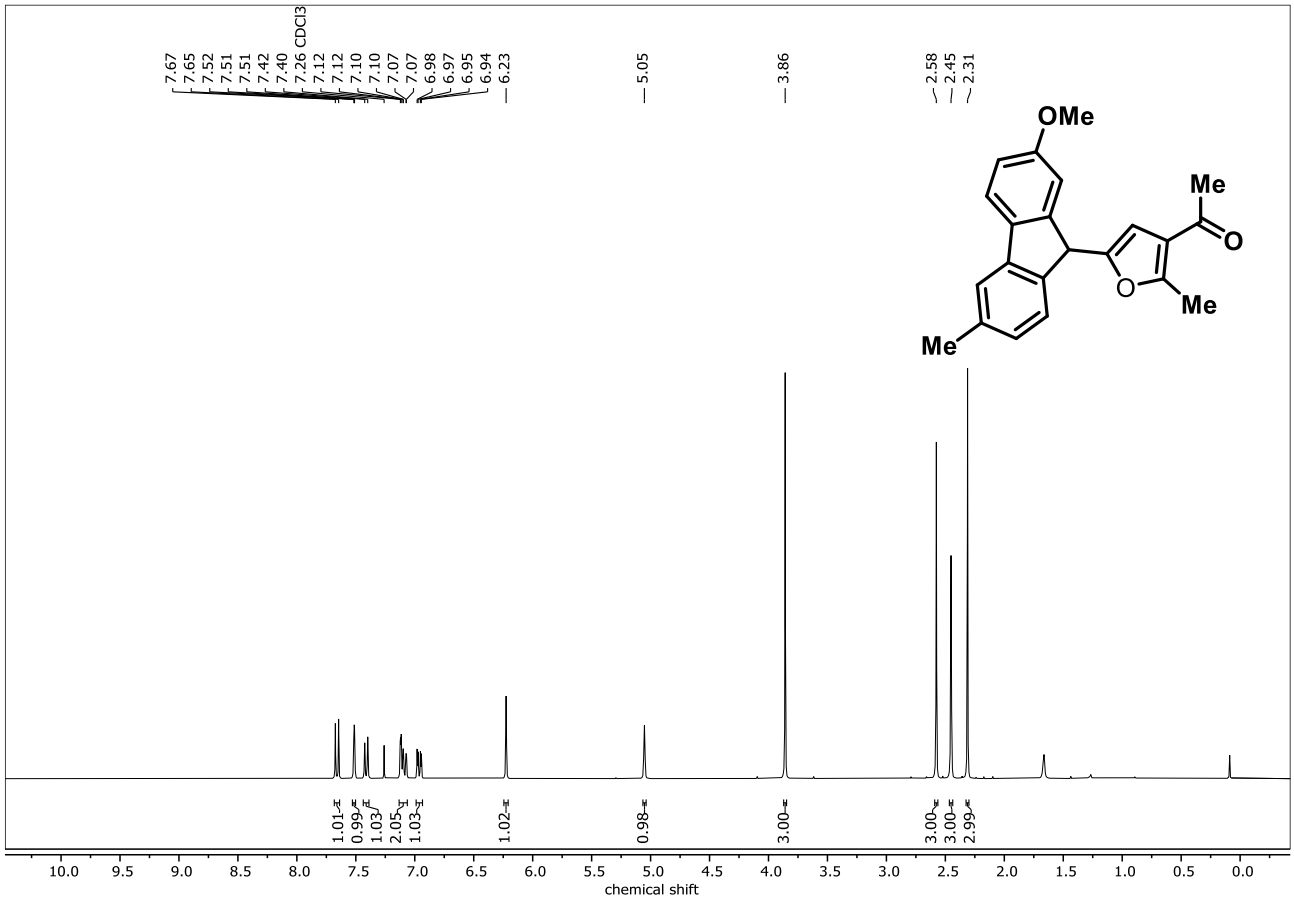
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3i



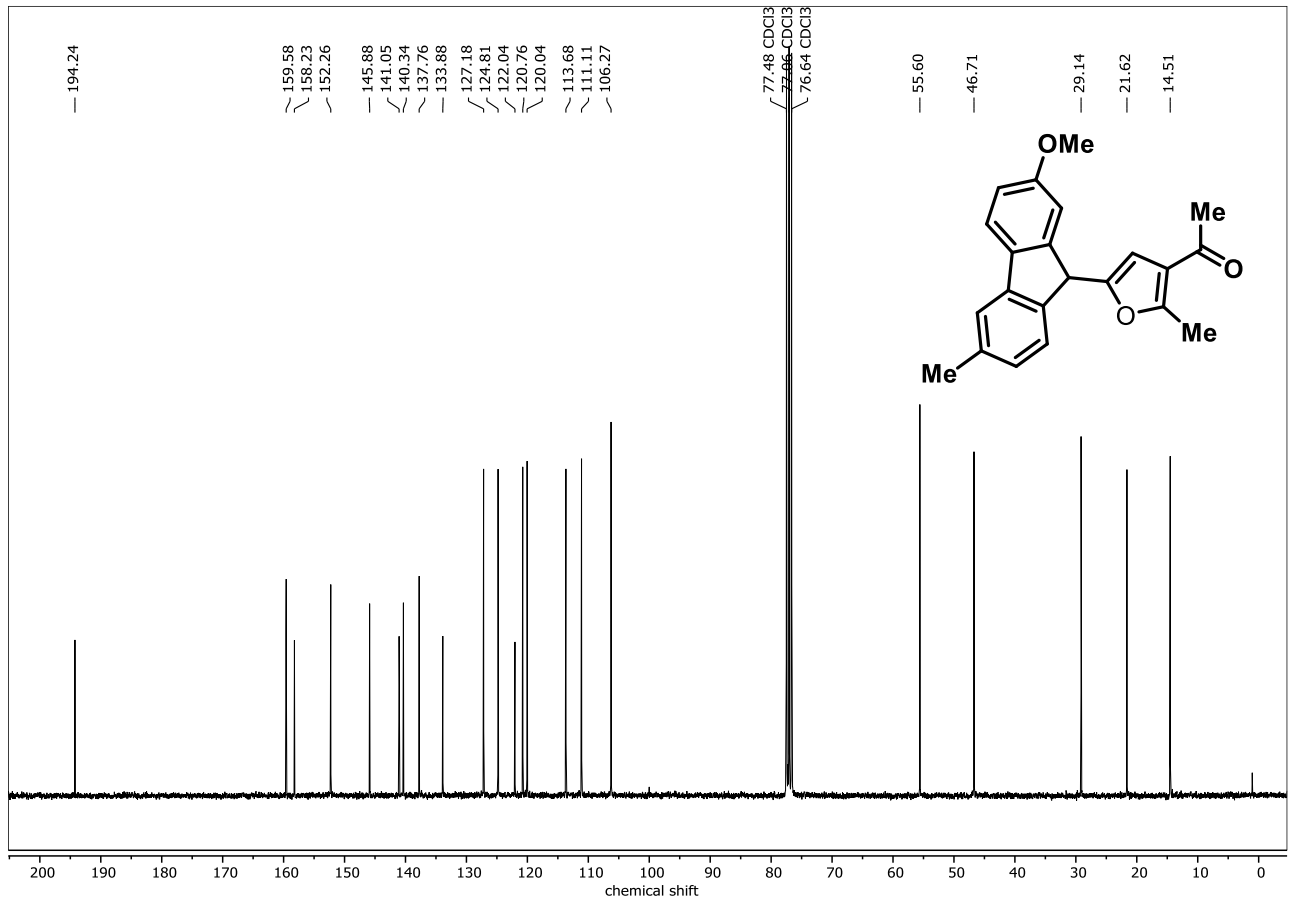
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3i



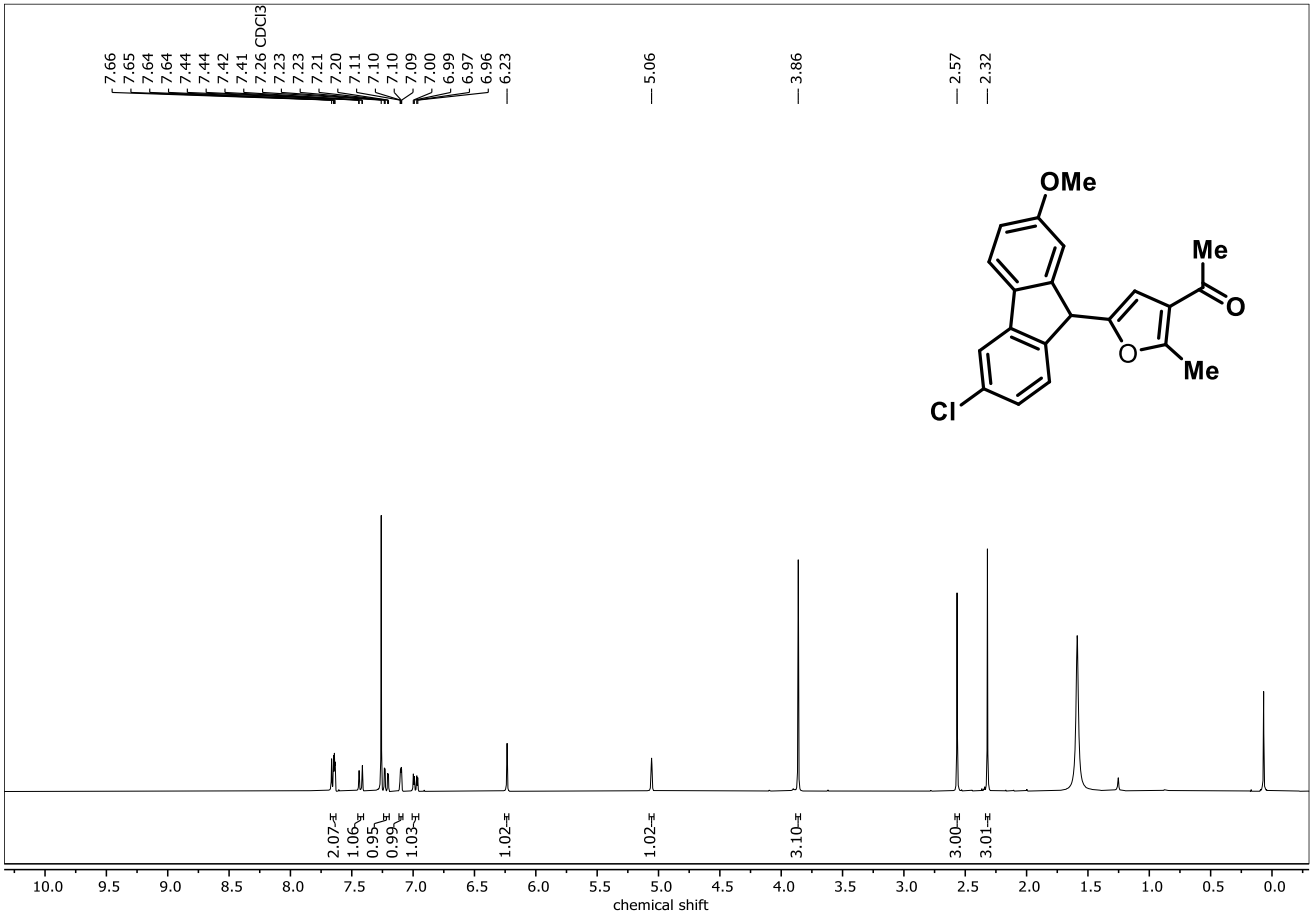
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3j



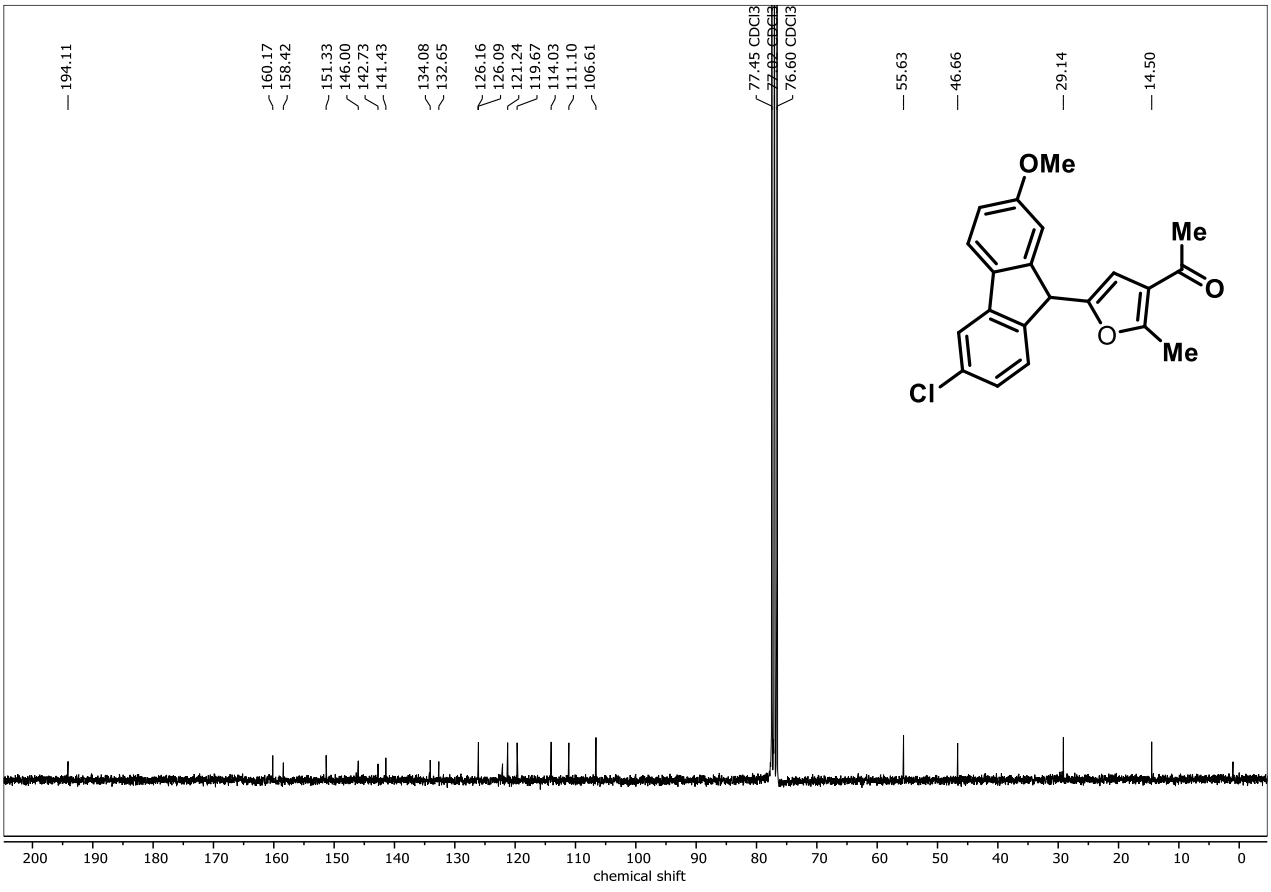
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3j



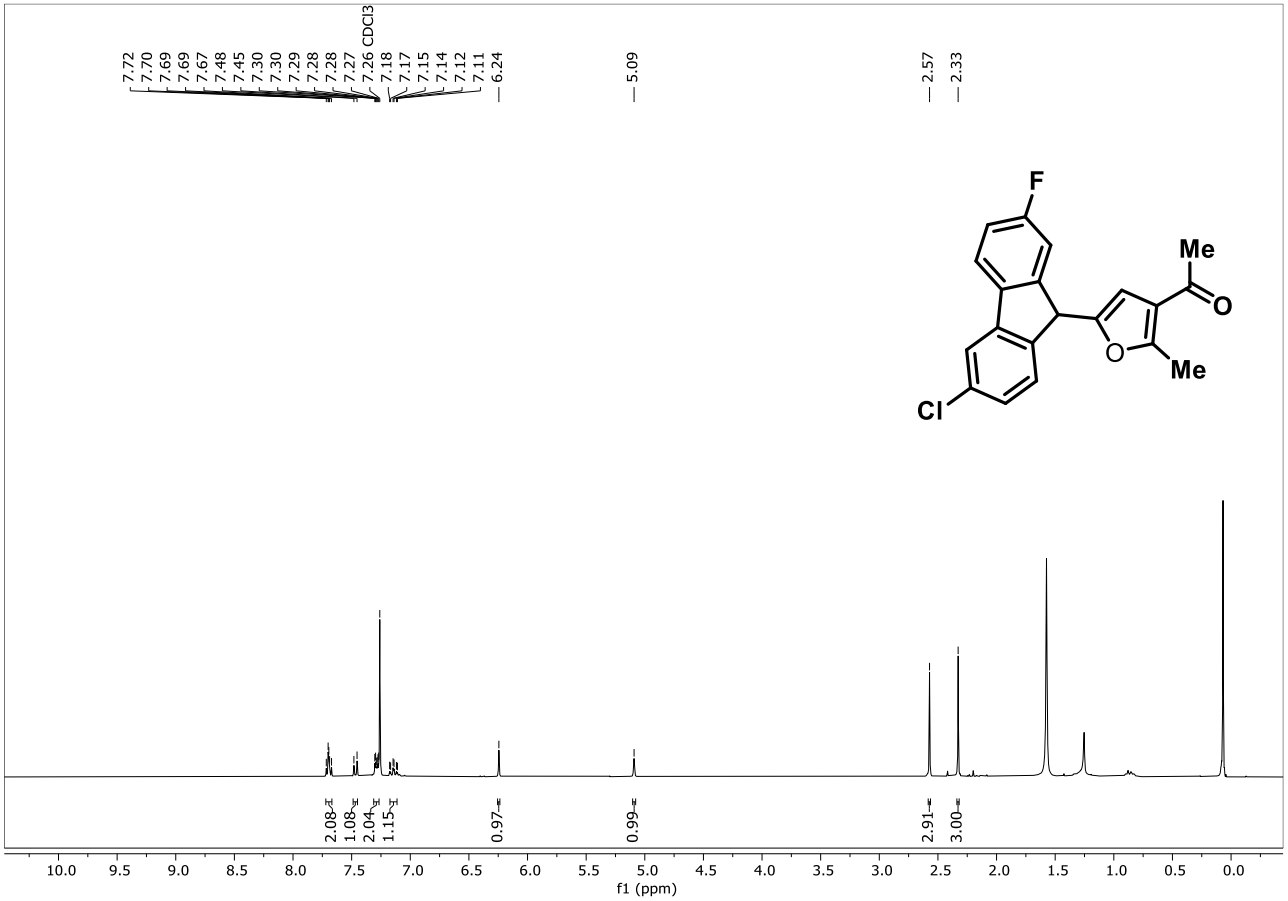
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3k



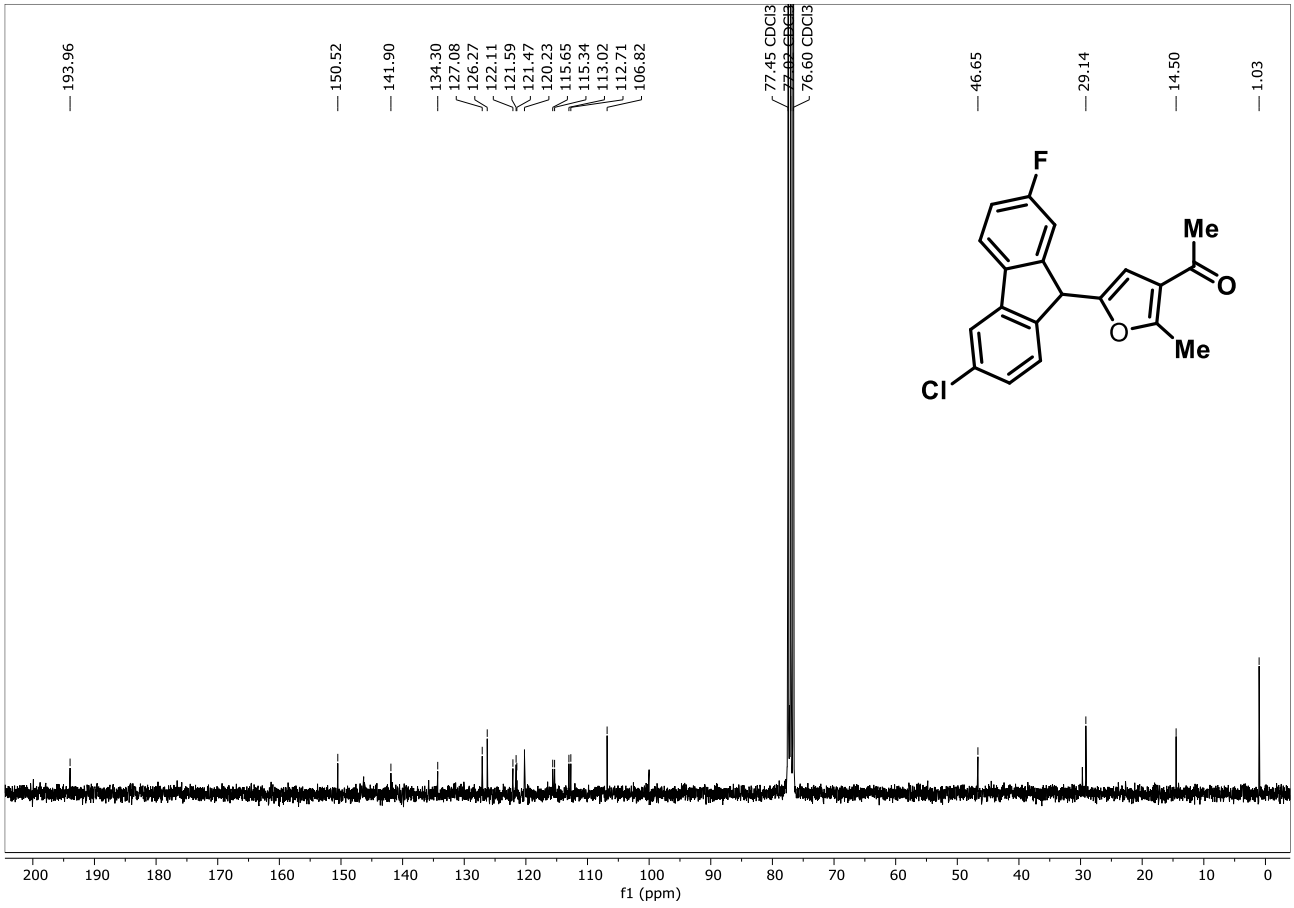
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3k



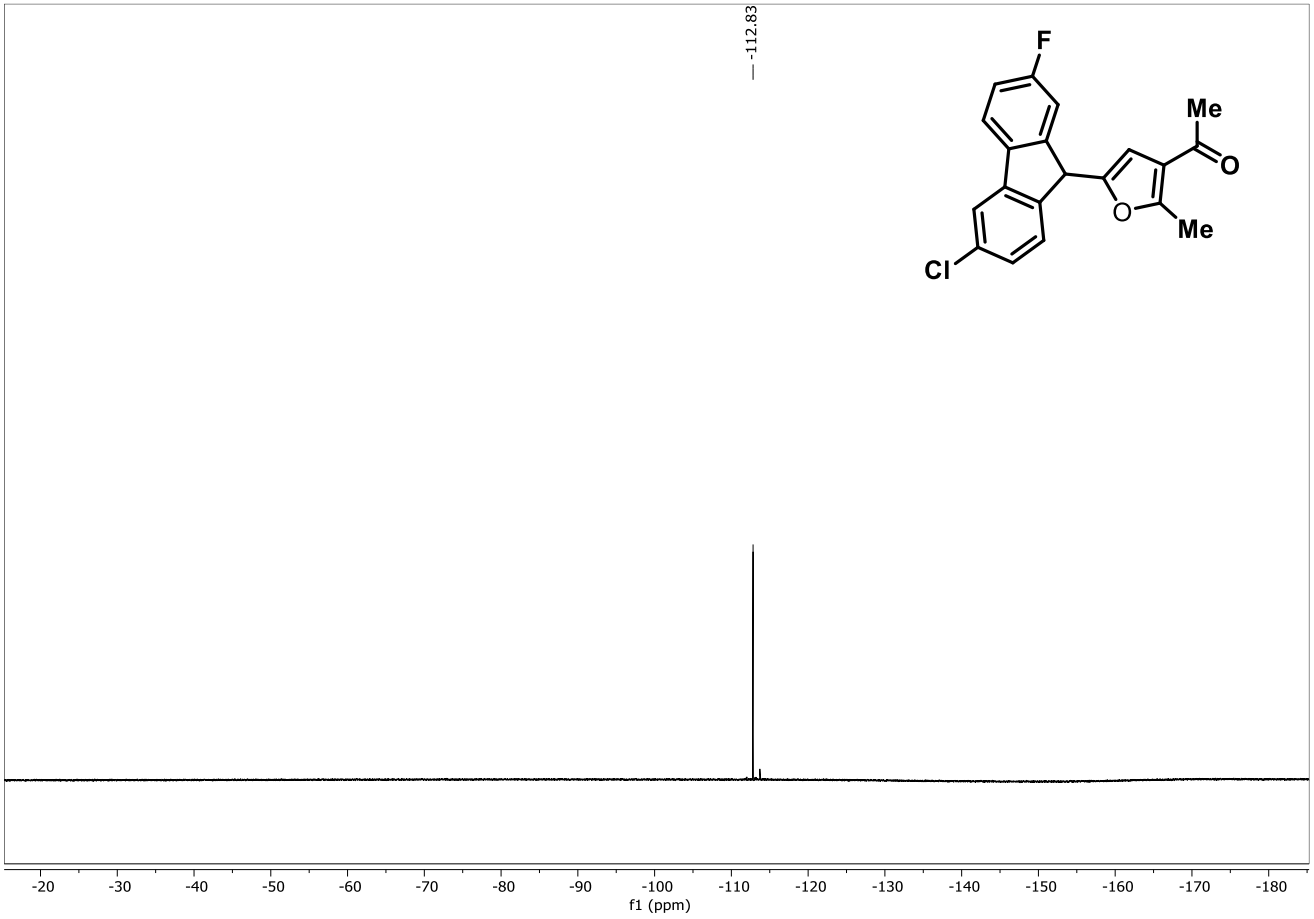
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3l



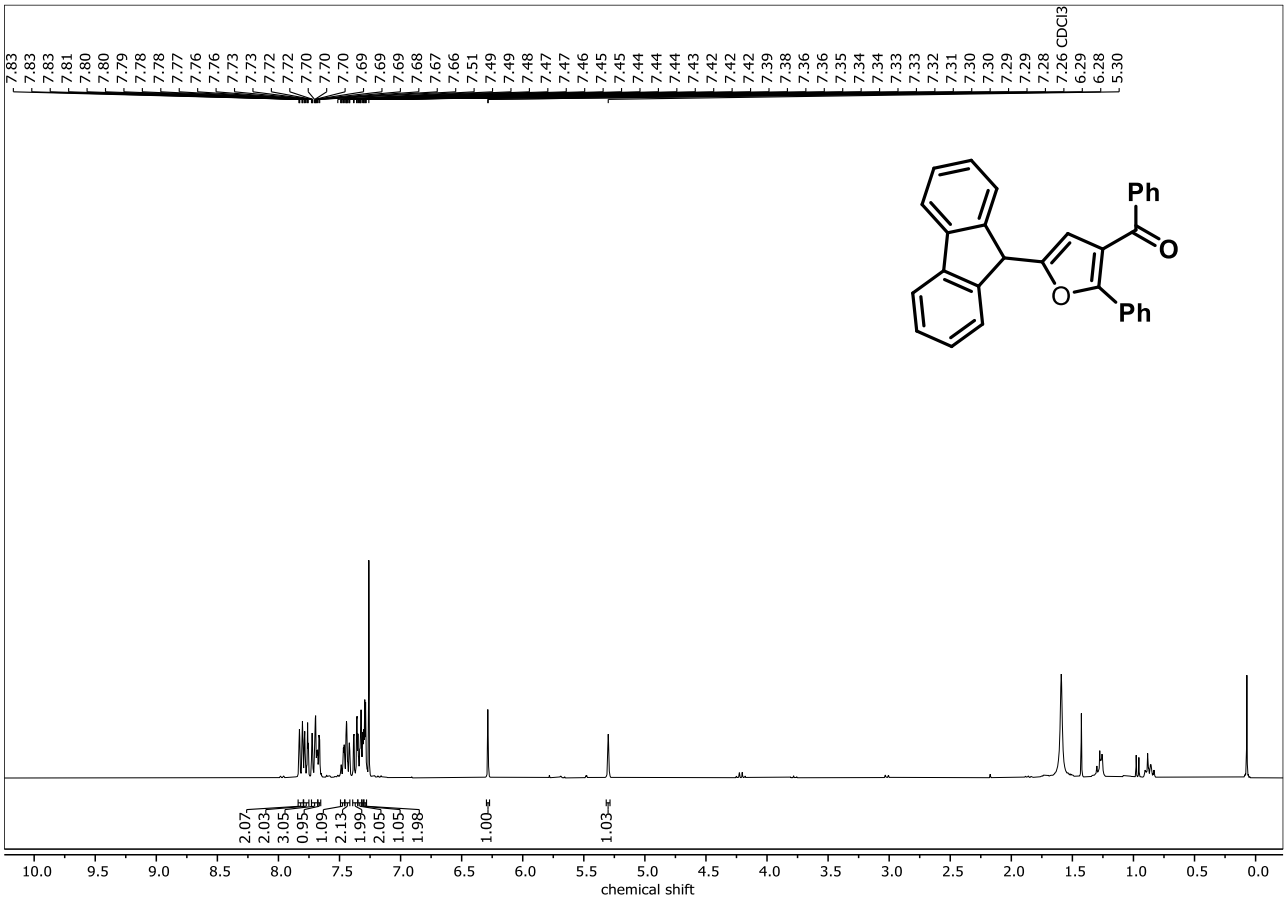
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3l



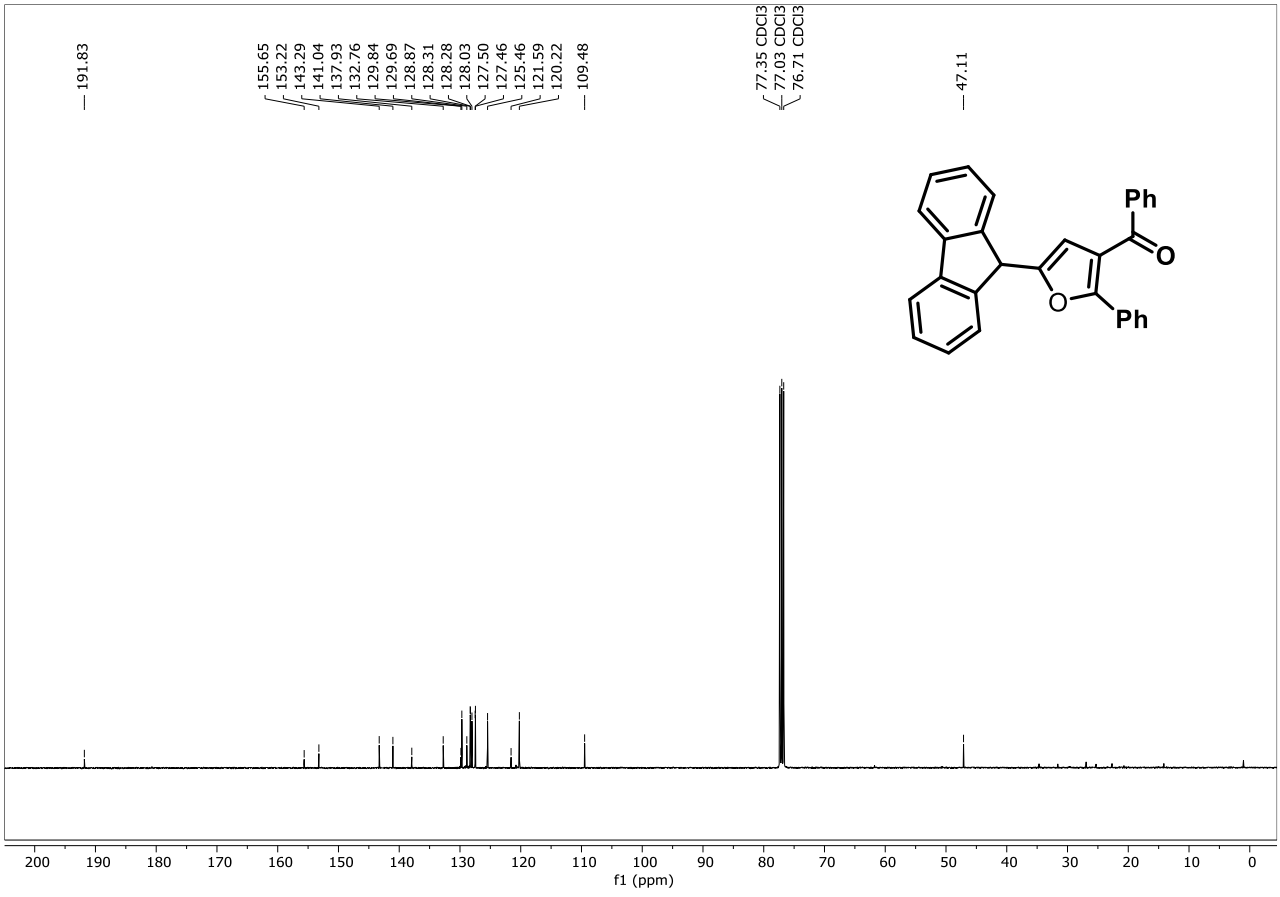
^{19}F NMR (282 MHz, $CDCl_3$) Spectrum of 3l



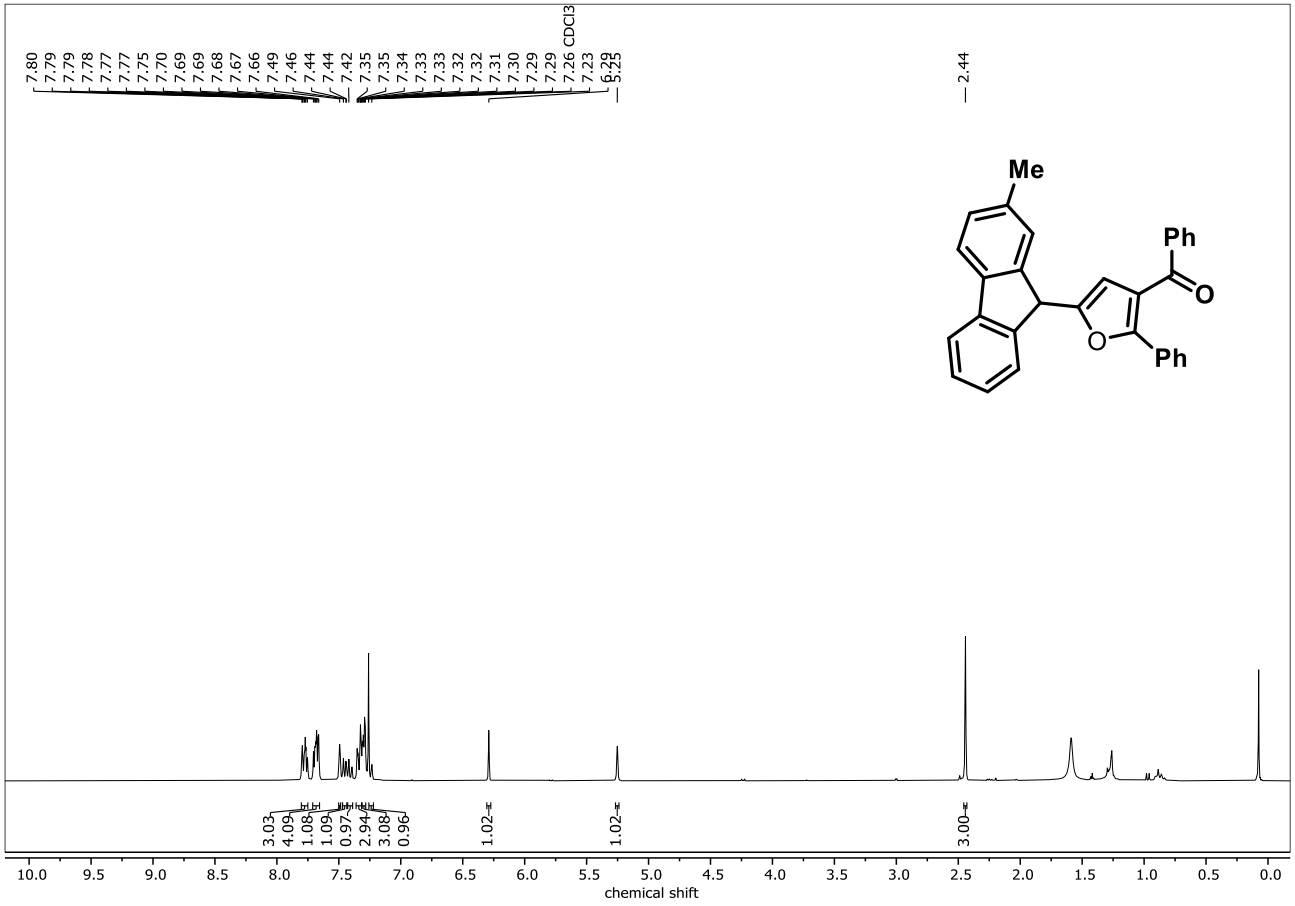
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3m



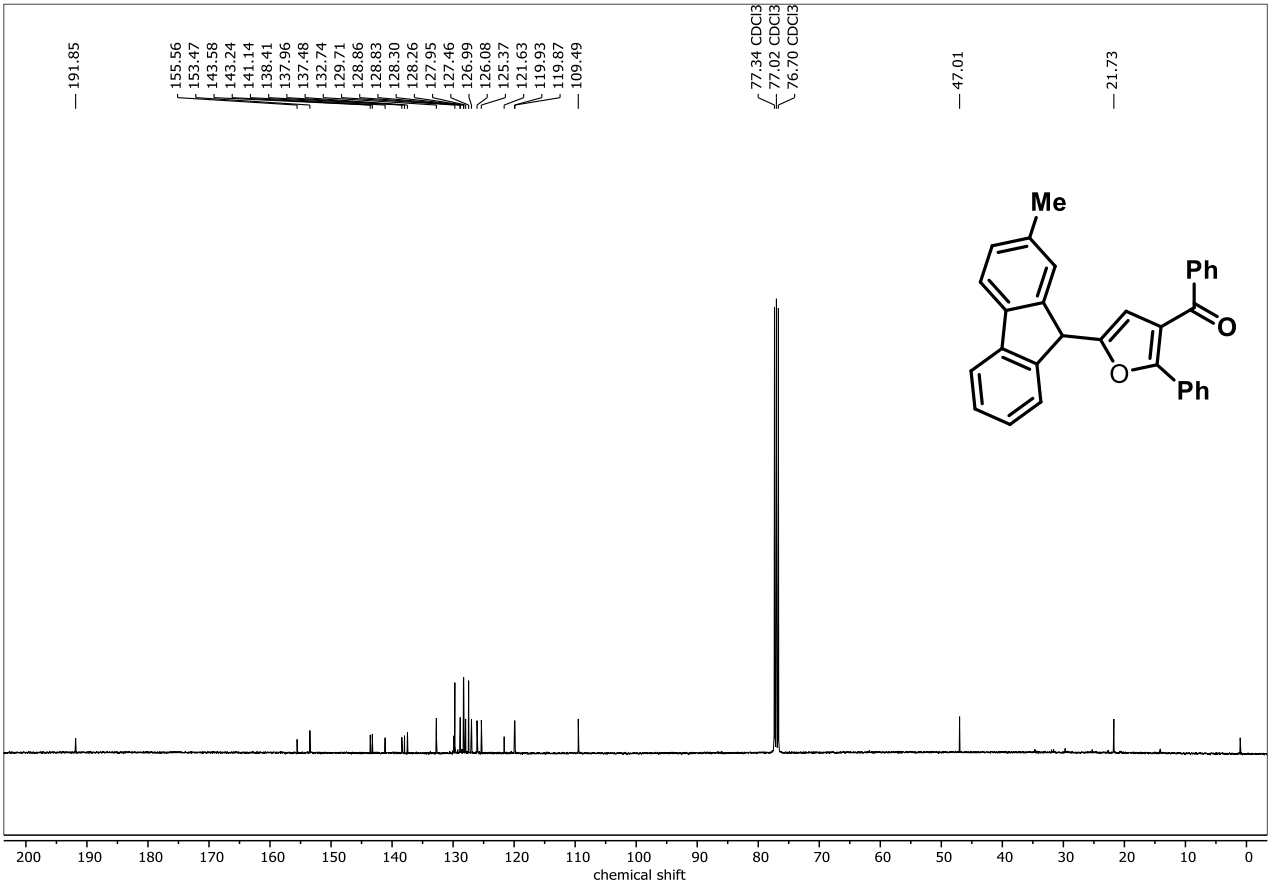
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3m



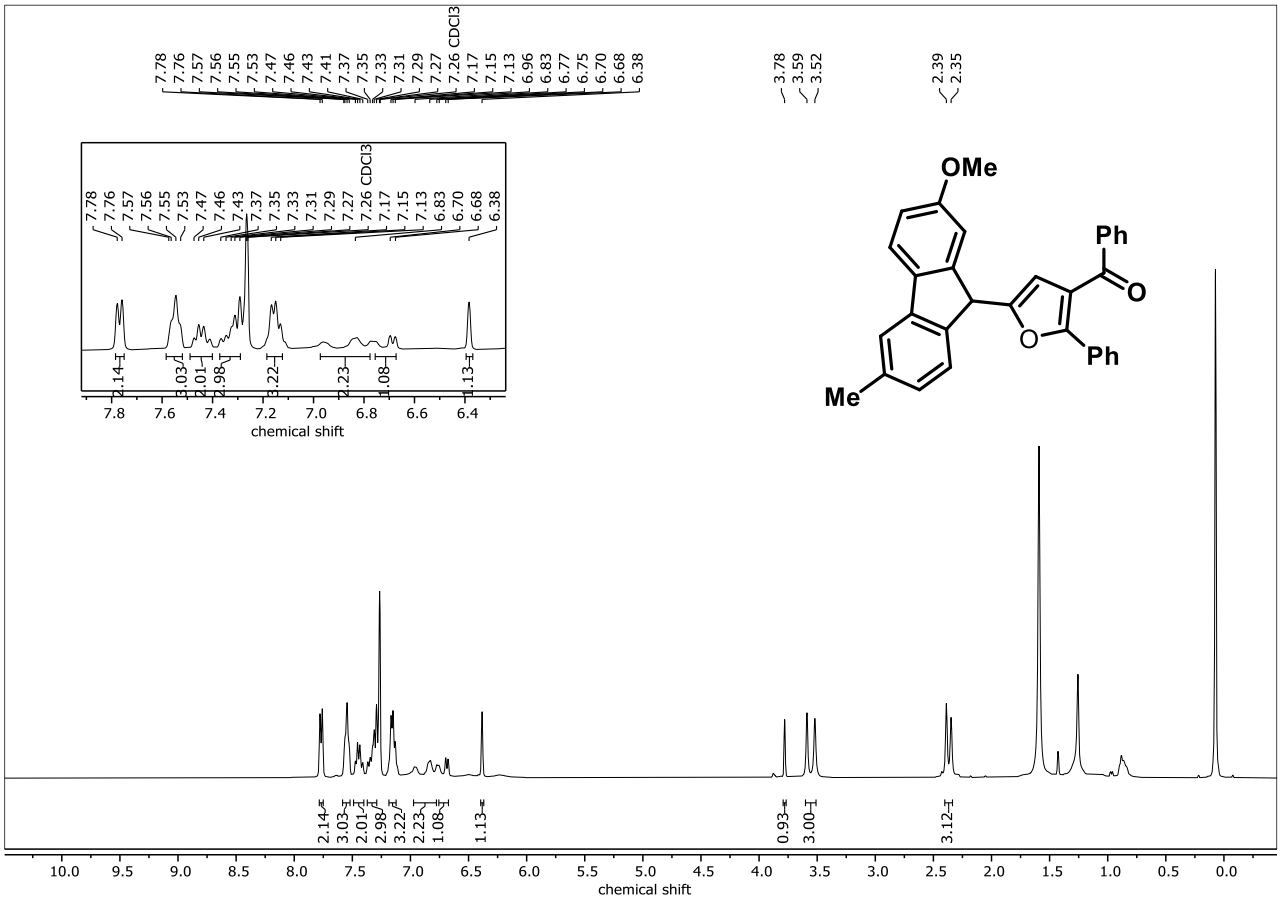
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of 3n



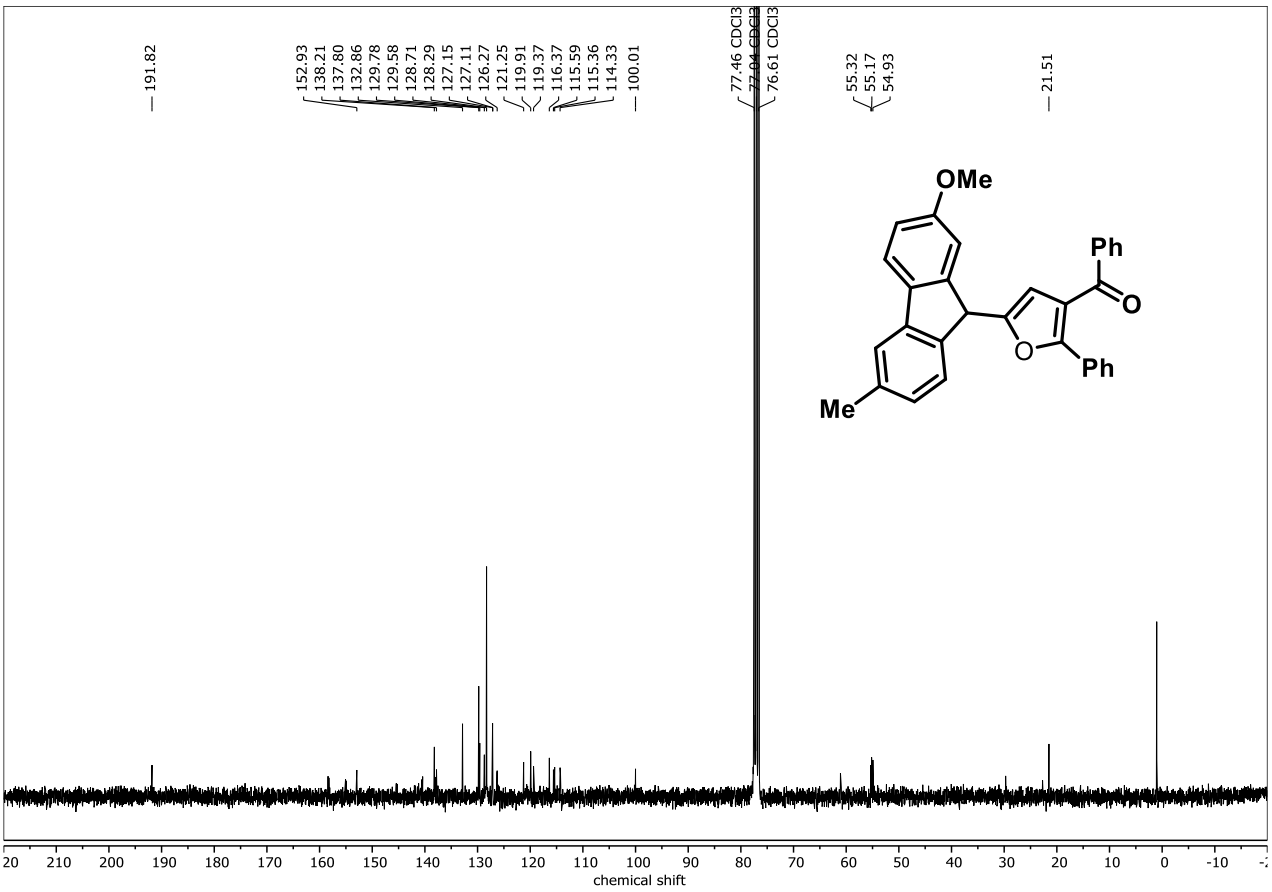
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3n



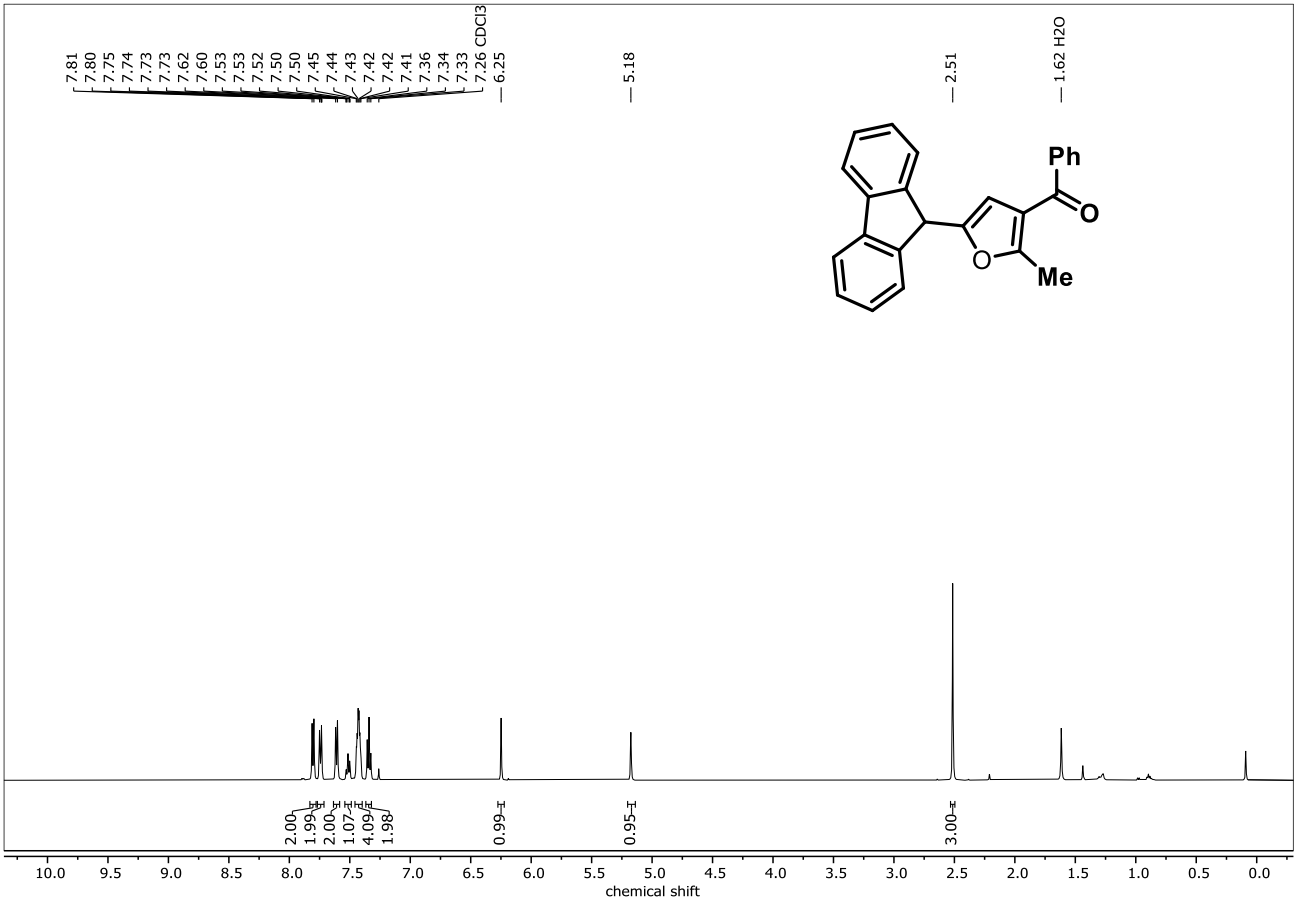
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of 3o



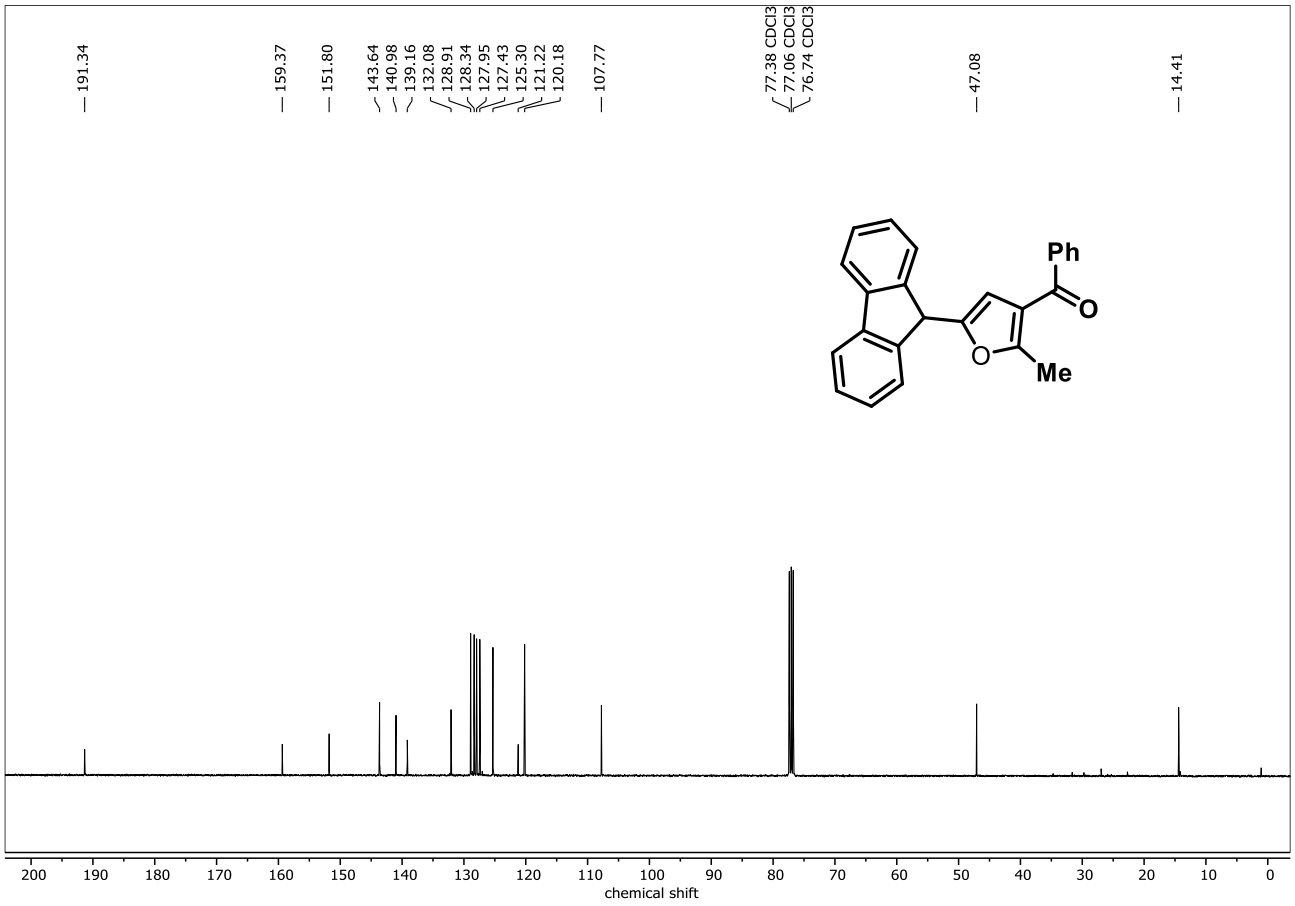
^{13}C{^{1}H} (75 MHz, $CDCl_3$) NMR Spectrum of 3o



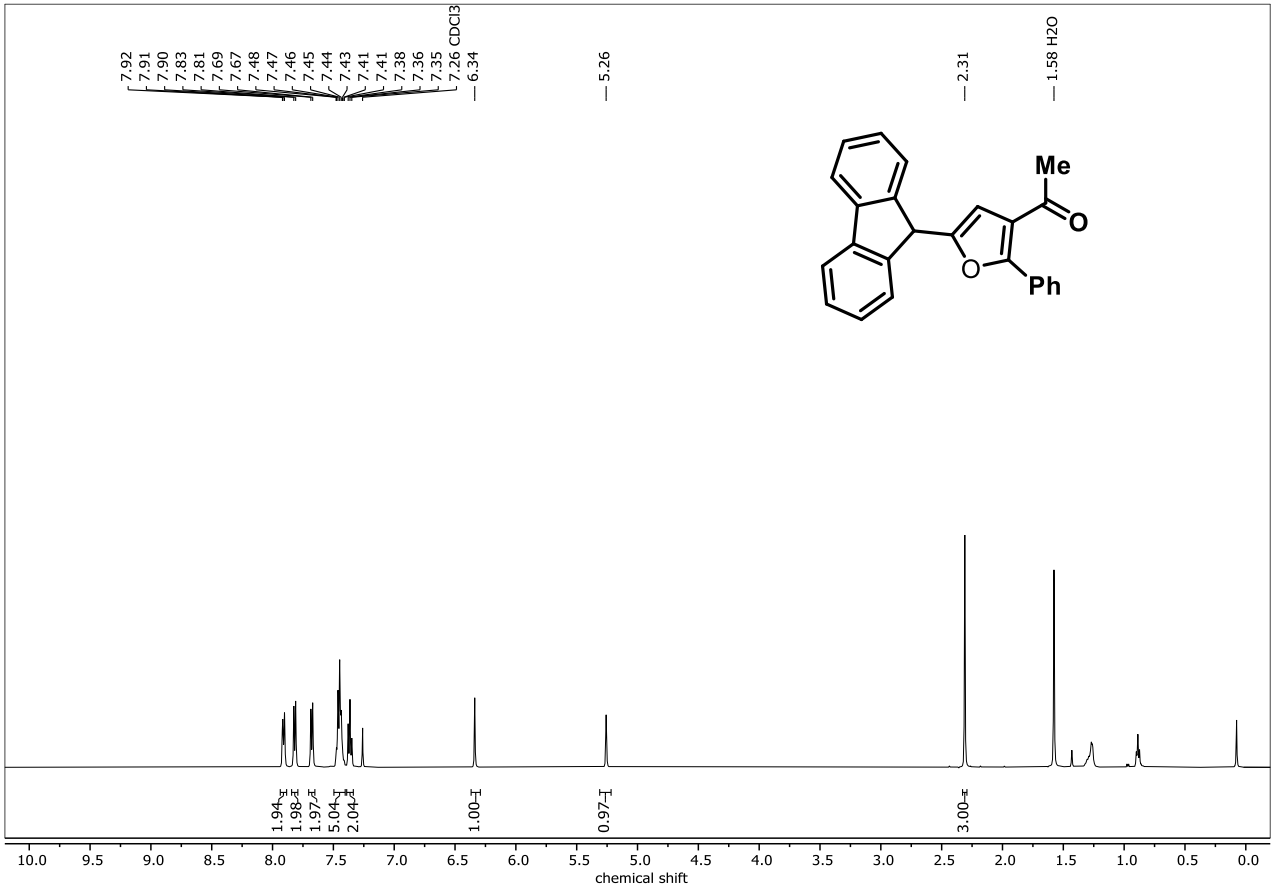
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3p$_a$



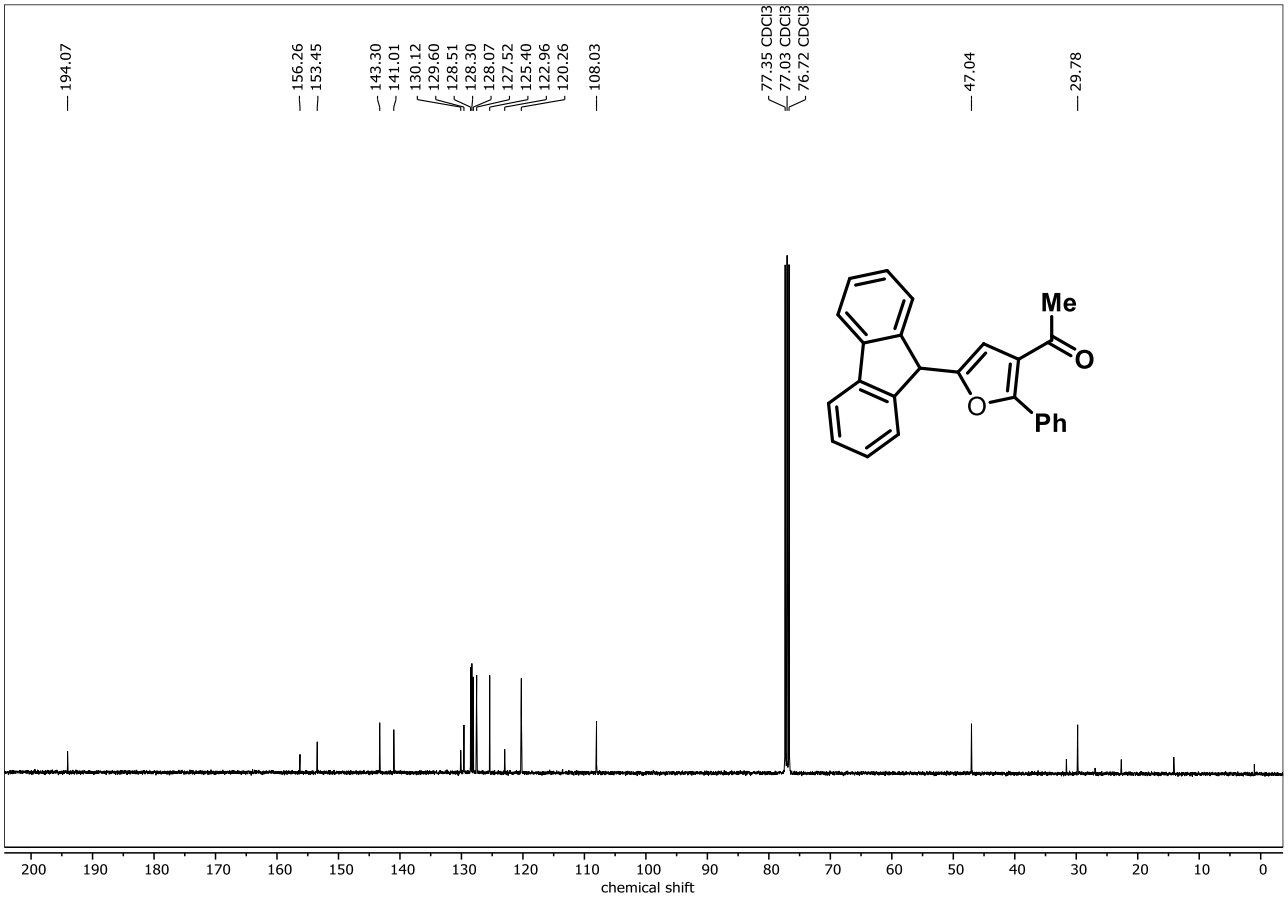
^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3p$_a$



^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of 3p$_b$



^{13}C{^{1}H} (101 MHz, $CDCl_3$) NMR Spectrum of 3p$_b$



Sample preparation and crystal structure determination for 3a

The single crystals of the compound 3a were obtained by slow evaporation from a dilute solution of 3a in dichloromethane and hexane at room temperature. The single crystal suitable for X-ray of compound 3a was mounted on the tip of a thin glass fiber with commercially available adhesive. The X-ray single crystal data collection of 3a crystal was performed at room temperature using a Bruker APEX III D8 Quest smart diffractometer, equipped with a microfocus and a sealed tube X-ray source with graphite mono-chromated Mo-Kα radiation (λ = 0.71073 Å). The data were integrated using the SAINT1 program, and the absorption corrections were made with SADABS.2 The structure was solved by SHELXS 20173 using the Patterson method and followed by successive Fourier and difference Fourier synthesis. Full matrix least-squares refinements were performed on F2 using SHELXL-20174 with anisotropic displacement parameters for all non-hydrogen atoms. All hydrogen atoms were fixed geometrically by HFIX command and placed in ideal positions. All calculations were carried out using SHELXS-2017, 3 SHELXL-2017, PLATON v1.15,4 ORTEP-3v2,5 and WinGX system Ver-1.80.6 The data collection and the structure refinement parameters and crystallographic data for the compound are given in Table S1.

Table S1: Crystallographic Data and Structural Refinement Parameters for 3a (CCDC No – 2420729)

Empirical formula	$C_{20}H_{16}O_2$
Formula weight	288.33
Temperature/K	273
Crystal system	triclinic
Space group	$P\bar{1}$
a/Å	5.1390(2)
b/Å	12.3696(5)
c/Å	12.8834(5)
α/°	106.286(1)
β/°	96.791(1)
γ/°	101.912(1)
Volume/Å^3	755.53(5)
Z	2
μ/mm^{-1}	0.081
F(000)	304.0
Index ranges	$-7 \leq h \leq 7$, $-19 \leq k \leq 18$, $-20 \leq l \leq 19$
Radiation	MoKα (λ=0.71073)
2Θ range/°	6.828 to 67.154
ρ_{calc} (g/cm^3)	1.267

ORTEP diagram of the crystal structure of 3a.